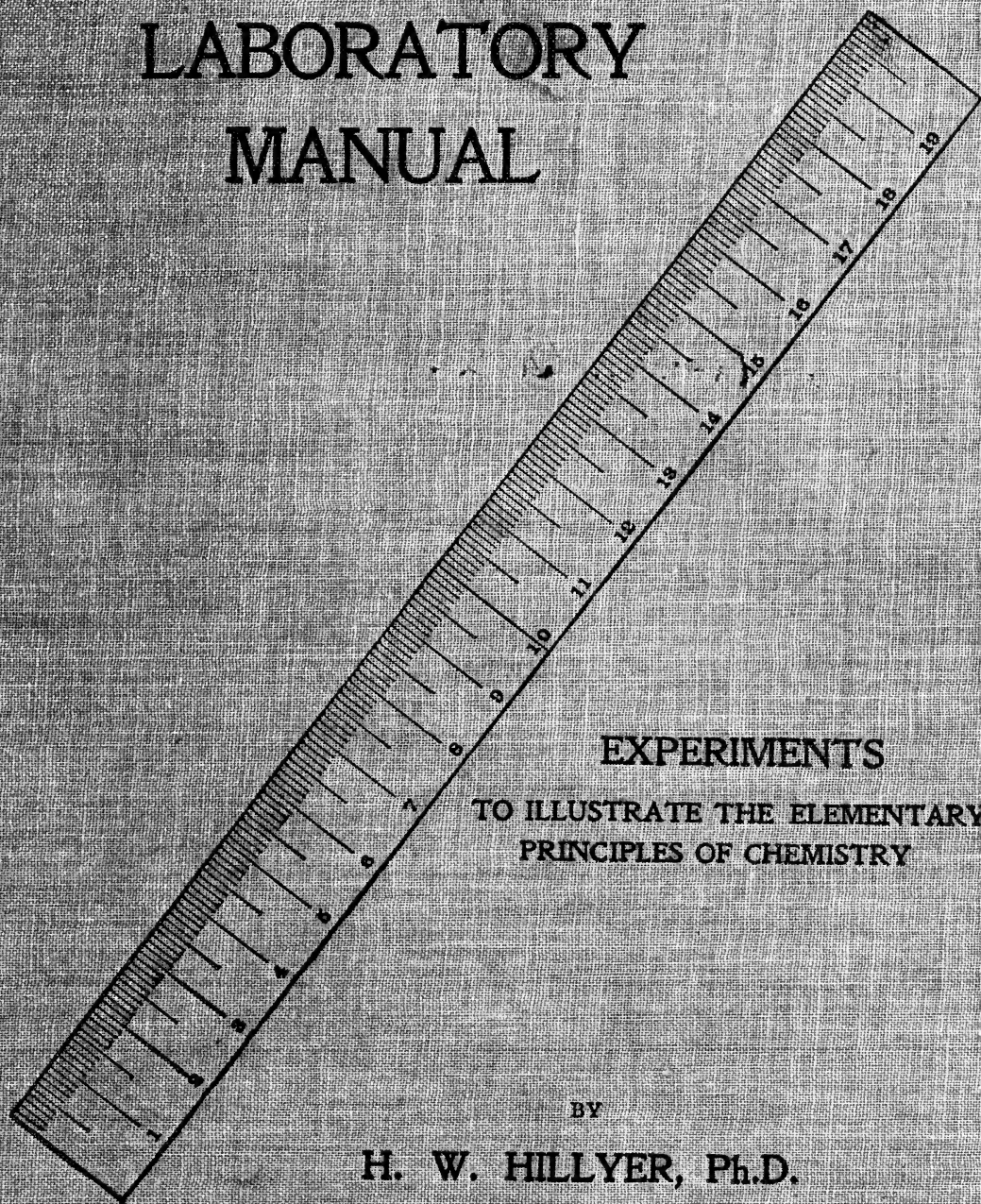


UC-NRLF



5B 35 431

LABORATORY MANUAL



EXPERIMENTS
TO ILLUSTRATE THE ELEMENTARY
PRINCIPLES OF CHEMISTRY

BY

H. W. HILLYER, Ph.D.

LIBRARY
OF THE
UNIVERSITY OF CALIFORNIA.
GIFT OF

R. S. Norris. Santa Maria
Received *Feb. 1900* *colib*

Accession No. *78217* . Class No. .

LABORATORY MANUAL

•The M Co. •

LABORATORY MANUAL

EXPERIMENTS

TO ILLUSTRATE THE ELEMENTARY
PRINCIPLES OF CHEMISTRY

BY

H. W. HILLYER, PH.D.

ASSISTANT PROFESSOR OF ORGANIC CHEMISTRY IN THE
UNIVERSITY OF WISCONSIN



New York

THE MACMILLAN COMPANY

LONDON: MACMILLAN & CO., LTD.

1899

All rights reserved

Copyright, 1899,
By THE MACMILLAN COMPANY.

78217

Norwood Press
J. S. Cushing & Co. - Berwick & Smith
Norwood Mass. U.S.A.



CONTENTS

	PAGE
MANIPULATION	4
TO THE STUDENT	26

PART I

PREPARATION AND PROPERTIES OF THE ELEMENTS AND THEIR COMPOUNDS

TYPICAL ELEMENTS :

HYDROGEN	30
CHLORINE	38
OXYGEN	46
NITROGEN	60
CARBON	76
SODIUM AND POTASSIUM	86
CALCIUM, STRONTIUM, AND BARIUM	92
HALOGENS	94
SULPHUR	100
PHOSPHORUS	110
ARSENIC, ANTIMONY, AND BISMUTH	120
BORON AND SILICON	126
MAGNESIUM AND ZINC	134
SILVER, COPPER, AND MERCURY	136
TIN AND LEAD	142
NICKEL AND COBALT	146
IRON	148
ALUMINUM	152
CHROMIUM	154
MANGANESE	158

PART II

VERIFICATION OF QUANTITATIVE LAWS

	PAGE
CONSTANT PROPORTIONS	162
MULTIPLE PROPORTIONS	168
COMPOSITION OF HYDROGEN CHLORIDE	170
COMPOSITION OF AMMONIA	174
COMPOSITION OF WATER	178
HYDROGEN EQUIVALENT OF ZINC	180
OXYGEN EQUIVALENT OF ZINC	182
VAPOR DENSITY AND MOLECULAR WEIGHT	184
MOLECULAR WEIGHT BY CHEMICAL MEANS	190

APPENDIX

CALCULATION OF GAS VOLUMES	196
TENSIONS OF AQUEOUS VAPOR	
20 CENTIMETRE SCALE IN MILLIMETRES }	200



INTRODUCTION

THIS book is written for the use of college students of general chemistry. In attendance on lectures in general chemistry there are students who may be put in three groups, although these groups are not distinct. For those who are beginning the study, the required experiments have been arranged. In this part, experiments on a few typical elements have been first introduced that their properties may illustrate the fundamental ideas of chemistry. These are followed by experiments on the remaining common elements so arranged as to lead from the study of elements of simple relations to those of more complex relations, while keeping together as far as possible those elements of similar chemical properties.

Along with these required experiments a few others have been introduced, which are for the most part exercises in preparing substances of such marked physical properties that their purification and identification require no knowledge of analysis.

These Elective Exercises are for the profitable employment of those to whom, from previous training, a part of the required work is already familiar, or of

those who for some special reason find time for more than the regular work.

The quantitative experiments of Part II are designed more especially for those students who have had, in their preparatory schools, a good laboratory course but are not far enough advanced to be passed in collegiate general chemistry. These experiments are intended to replace for them entirely or in part the other laboratory work.

MANIPULATION

GLASS TUBING

Cutting.—To cut glass tubing into lengths, lay the tubing on the desk and with a triangular file make a vigorous cut on one side, then, taking the tubing in the hands, with the thumbs close together and against the tubing directly opposite the file cut, pull and at the same time slightly bend the glass, when it should break with a clean fracture at right angles to the length of the tube. Tubes of a diameter of more than one centimetre may need to be filed rather deeply, and perhaps also have a slight groove filed all around the tube.

Bending.—In bending glass tubing, it should be heated for a length of six or seven times the diameter of the tubing, that a regular curved turn may be made, and that the bore of the tube may not be contracted. This is accomplished by heating a short portion and making only a part of the bend, then heating a neighboring short portion and making a second part of the bend, and so on until the bend is complete. This method is necessary only when a flat flame is

not available. A flat flame may be made by using the so-called "wing top" on the Bunsen burner, or by using an ordinary illuminating burner. The smoky flame of the latter is by many preferred to the clear flame, and is very good for use with soft glass. In any case the bending must not be attempted until the

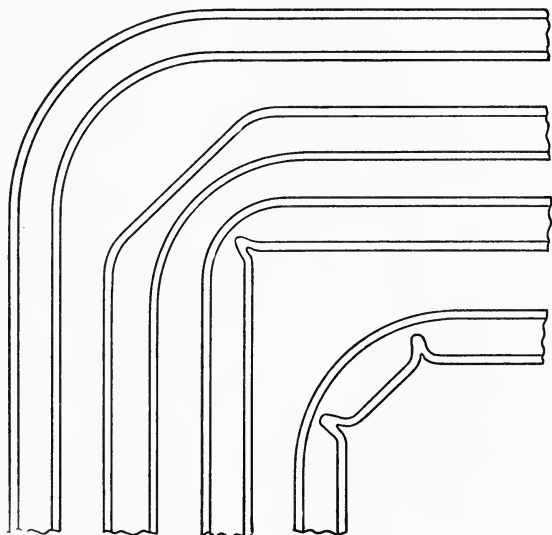


FIG. 1.

glass is quite thoroughly softened by the heat so that very little force is required in the bending. The tube must be constantly rotated while in the flame, that it may be heated on all sides alike. In the cut is shown a properly made bend, and three of the more common styles of badly made bend. In all of these the bore of the tube is contracted, and the last and next to the



last pieces are very likely to be broken if they are heated, on account of the thick lumps of glass made by bending the tube with too short a turn. All bends in a piece of tubing should be made in the same plane, so that the finished piece of apparatus will lie flat, and on that account not be so liable to be broken.

Drawing.—To draw out a tube so as to contract its bore, a portion of the tube is heated until it is quite thoroughly softened, the end portions of the tube being held in the hands. It is then taken from the flame and stretched until the heated part is contracted to the desired degree. To make a glass jet, the tube thus drawn need only be scratched with a file at the narrow part and broken off.

Sealing.—To seal a tube at one end, it may be drawn out as indicated above, then heated somewhat more at

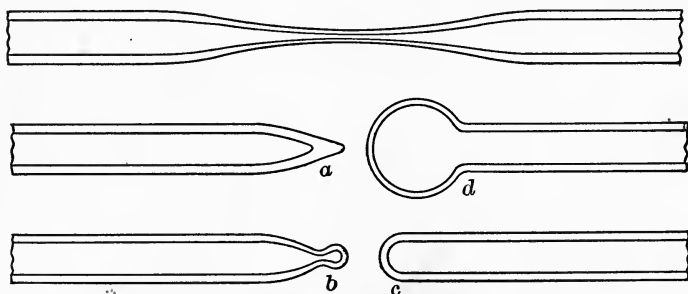


FIG. 2.

the narrow part, and again drawn until the bore is entirely closed and the tube melted apart. By holding

the closed end in the flame, the heated parts contract and thicken so that the parts become more uniform. For many purposes a seal of this kind is good enough, but when the sealed end is to be again heated, a hemispherical closure with walls of even thickness is to be preferred, and may be made in the following way. First, heat the little solid tail of glass (*a*) at the end of the closure, made as described above, until it is very hot; then remove from the flame, put the open end in the mouth, and force air into it in successive short impulses. Heat again, if necessary, and again blow as before until the solid tail is expanded into a thick, small bulb (*b*). Now heat the whole conical end and blow as described until the end is forced out tangent to the sides of the tube as in (*c*).

Small bulbs may be made at the end of a tube, after closing it by the operation just described, by heating, before the final blowing, a larger portion of the tube. Bulbs are, however, rarely necessary in elementary work.

Smoothing edges.—The sharp edges made by cutting glass tubing should be smoothed so that the rubber stoppers and connectors may not be cut in putting apparatus together. This may be done by holding the end of the tube in the flame and rotating it there until the rough edges are rounded by fusion.

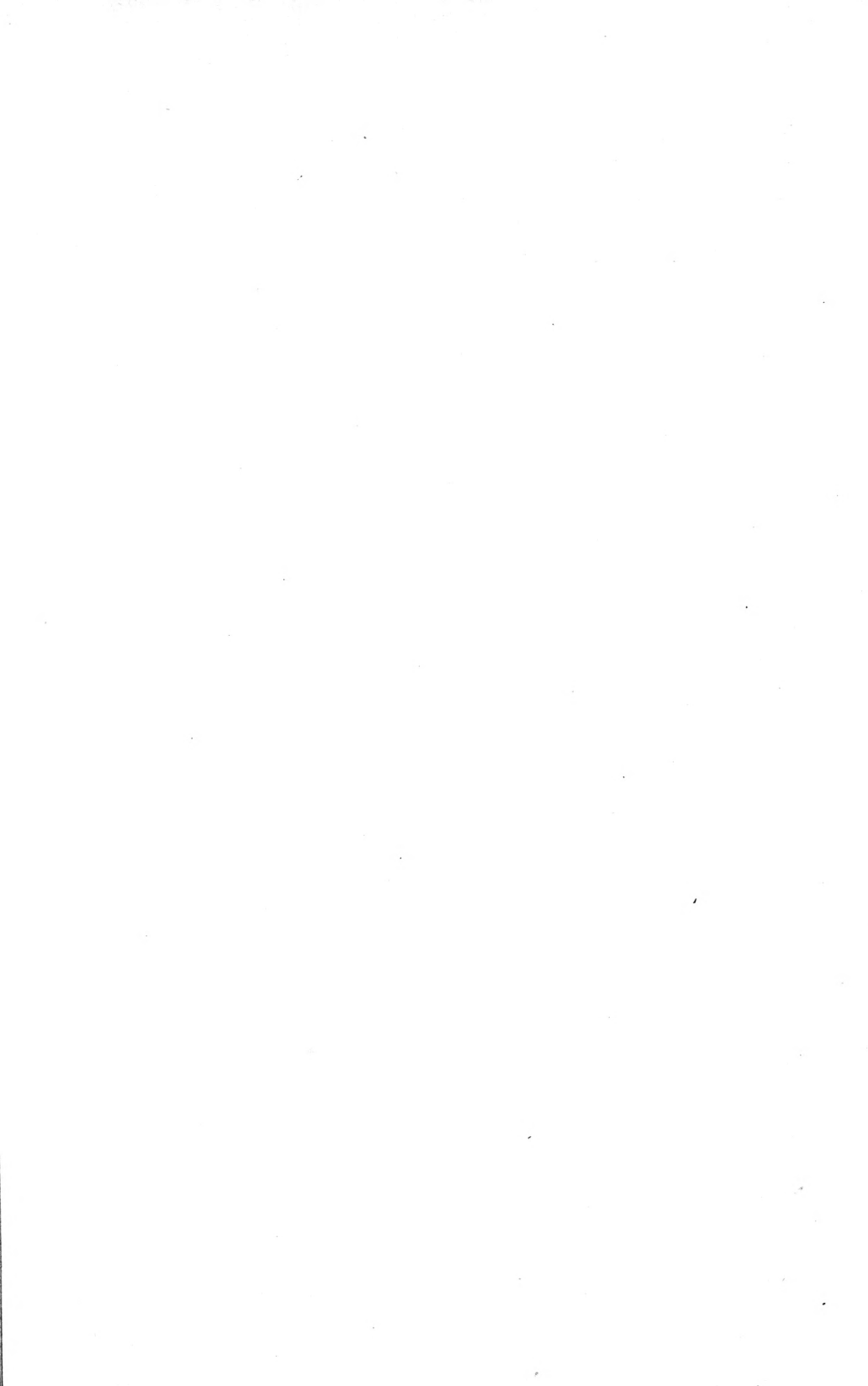


Corks.—Corks before being used as stoppers should be softened with a cork press, or by rolling them on the desk top with a board, or even on the floor under the foot if care is taken to well wash them afterward.

Corks may be perforated for the insertion of glass tubing by means of a coarse round file, using the handle end to perforate the cork, and the rough end to file out the hole thus made until it is of the right size to snugly fit the glass tubing. Special cutters are also used for perforating corks. They are brass tubes of various sizes sharpened at one end so as to have a circular cutting edge. To make a smooth hole in which the tubing will fit tightly the cutter should not be forced as a punch through the cork, but pressed lightly against the cork as it is turned back and forth on its axis and cuts its way through.

Cork cutters may also be used to cut holes through rubber stoppers, if they are dipped in vaseline, and if care is taken to work the vaseline into the cut by taking the cutter out and putting it in frequently during the operation, which should not be forced. Strong solutions of an alkali may also be used in cutting holes in rubber stoppers.

Stoppers should be so well fitted to flasks and tubing that the joints shall be tight without the use of paraffin, sealing wax, or other makeshifts. Experiments often fail on account of leaky joints in the apparatus.



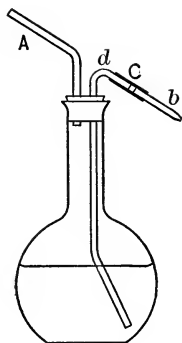


FIG. 3.

EXERCISE.—To put these principles in practice, make the wash bottle shown in the cut (Fig. 3). It will frequently be needed, and its parts may be used in making other apparatus. (*A*, *b*) and (*d*) are of glass. (*C*) is a connector of rubber tubing.

Show the finished apparatus to an instructor for criticism.





PURIFICATION

Purification by washing. (1) **Decantation.** — In this method the insoluble portion of a mixture is freed from the soluble portion by bringing the soluble part into solution, letting the insoluble part settle, and pouring off, “decanting,” the solution of the soluble part. To continue the washing, the residue is again mixed with the solvent, again allowed to settle, and again freed from the liquid by decanting. Sand, for example, can by this means be easily freed from blue copper sulphate, which is soluble.

(2) **Filtration.** — In the second method the insoluble solid is brought upon a filter. Water is then poured, or better, spirted upon it from a wash bottle (Fig. 3). When this water has all run off, water is again applied and allowed to run off completely. The same operations are repeated until the solid is freed from soluble matter. Why is it best to let the water run off completely each time before putting on more? This method is used especially with precipitates.

The filter paper used should be cut in circles of such a size that when folded and placed in the funnel it fails to reach the edge of the funnel by from 5 to 10 mm.

Filter papers which project above the edge of the funnel are washed with very great difficulty, because the projecting edge continually draws up the solution by capillary action. For all ordinary use there is only one way to properly fold a filter paper. Fold it in halves on a diameter and then fold it in quarters. Open up on the curved edge and then place in the funnel with one thickness of paper on one side and three thicknesses on the other. Properly made funnels have an inner angle of 60° , and

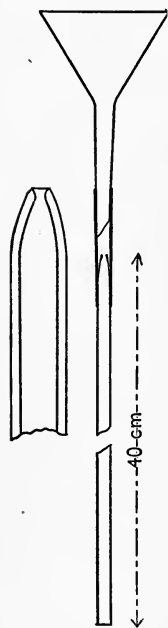


FIG. 4.

a filter folded as indicated will fit smoothly against the sides of the funnel. — Why? — If the paper does not fit tightly against the sides of the funnel, it should be adjusted to fit, since then only can it be washed rapidly and thoroughly.

In filtering, the vessel receiving the filtrate should be so placed that the liquid will not fall into it in drops, but will run down the side of the vessel. This is to avoid spattering.

In some cases the liquid runs off slowly, and filtering and washing require much time. This difficulty may be avoided to some extent by extending the stem of the funnel with a tube slightly contracted at one end. This contracted end is pushed into a rubber connector attached to the tube of the funnel. The contraction in the tube causes the tube to

fill with the filtrate, and the atmospheric pressure is thus reduced on the lower side of the filter. The tube should not be more than 40 cm. long. To make this device of any value, the filter paper must be well fitted to the funnel.

In filtering and transferring liquids from one vessel to another, the liquid frequently runs down the outside of

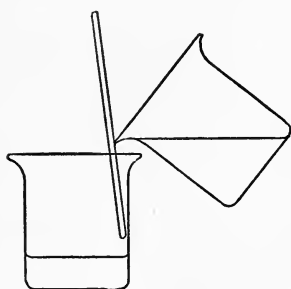


FIG. 5.

the vessel from which it is being poured. This may be avoided by resting a solid glass stirring rod against the outside of the vessel from which it is being poured at the point where the liquid is to flow out. The liquid will generally follow the rod. It may be kept from running down the out-

side of the dish by slightly oiling the finger with vaseline and rubbing it upon the edge over which it is to flow.

Purification by crystallization. — **EXERCISE:** Dissolve 50 grammes of alum, $K_2Al_2(SO_4)_4$, and 5 grammes of copper sulphate, $CuSO_4$, in 75 c.c. of water by the aid of heat. Divide the solution into two parts. In one part suspend a string and set it away in a quiet place until next day. Then examine the crystals found and describe them as to shape, size, and color. Cool the other part of the solution rapidly under the hydrant, stirring it as it cools. When cool, filter off the crystals and wash them by pouring

water upon them and letting it run off, and then again pouring water upon them and letting it run off. What color has the solution? What color have the crystals? If they are not white, dissolve in a little hot water and again cool, filter, and wash. What have you learned by this experiment?

Purification by sublimation. — **EXERCISE:** Mix a little arsenic trioxide, As_2O_3 , with an equal quantity of sand. Put the mixture in a dry test tube and heat, not too strongly. Notice, on the cool parts of the tube, the crystals of arsenic trioxide freed from the sand with which it was mixed.

Purification by distillation. — **EXERCISE:** In a 125 c.c. tubulated retort put 50 c.c. of water and two or three

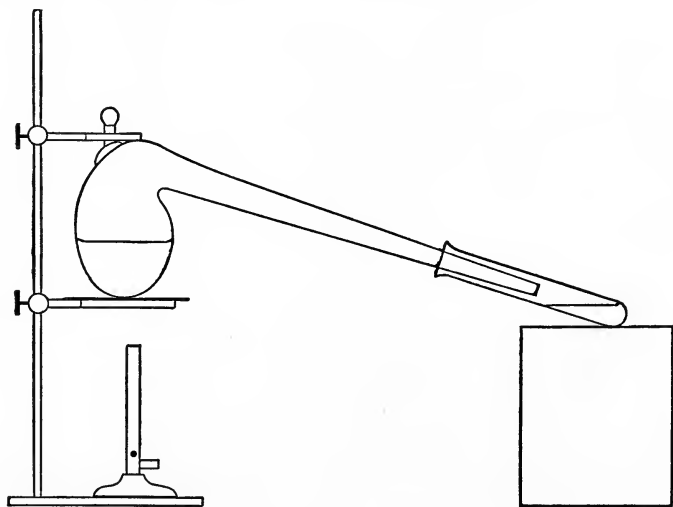


FIG. 6.

crystals of potassium permanganate, KMnO_4 . Heat the water to boiling and collect that which condenses in the neck of the retort in a small test tube used as a receiver. What color has the liquid in the retort? What in the receiver? What have you accomplished by the distillation?



TO THE STUDENT

IN beginning laboratory work for the study of any particular element, read that part of the *text book* which treats of the chemistry of the element. Before commencing the experiment, read through carefully the directions for performing it and also read the part of the text book dealing with the special point which the experiment illustrates. Do not begin an experiment until the directions have been carefully read and the purpose of the experiment grasped. The mere bringing of chemical substances into conditions under which they will react has less utility as a means of culture than most of the manual occupations.

Having the purpose of the experiment clearly in mind, read the directions again, date your note book, and perform the experiment, following directions carefully.

Write down in your note book the purpose of the experiment. Write down what *you observe*, not what you think you *should* observe. If you find that your observations do not seem to be in accordance with the text book or the statements of the lecturer, perform the experiment again with careful attention to the directions and then ask an instructor to explain any discrepancies which may be apparent. You will often

learn more in clearing away an apparent error than in performing an experiment which proceeds exactly according to expectation.

Before beginning the study of the next experiment ask yourself whether you have had any previous experiment at all like in principle to the one in hand, and make in your note book a short, clear statement of the points of similarity. Ask yourself also whether any general principle is illustrated by the experiment. The experiments frequently illustrate general principles and these ideas clearly grasped and clearly stated in your note book will be of great help in enabling the whole subject to be kept in mind.

To summarize; the study of an experiment, involves: (1) Reading of directions and text; (2) Understanding of the purpose of the experiment; (3) Thoughtful observation; (4) Truthful note making of the phenomena observed; (5) Comparison with other experiments; and (6) Drawing conclusions and searching for principles.

PART I

PREPARATION AND PROPERTIES OF THE ELEMENTS AND THEIR COMPOUNDS

TYPICAL ELEMENTS

1. Hydrogen from water. — Throw a piece of sodium, Na, the size of a small pea upon the surface of water contained in a large dish. Apply a lighted match to the sodium as it swims about. The hydrogen catches fire and burns with a yellow flame, due to the sodium vapor in the flame. *Do not put the face very close to the sodium as it floats on the water, as it may cause the alkaline liquid to spatter into the eyes. Be especially careful when the sodium has nearly disappeared.*

Pour some water on a piece of filter paper lying on the desk, and then place a small piece of sodium on the wet paper. Why is the action now different?

Try the same experiment, using potassium, K, instead of sodium. Note the resemblances and differences in the two cases. In these cases what is the source of the hydrogen?

Wrap a small piece of sodium in a piece of dry filter paper. Fill a six-inch test tube with water and invert it in a basin of water. Bring the mouth of the



tube as near the surface of the water in the basin as possible, and, holding the paper and sodium with a pair of forceps, quickly thrust them up into the mouth of the tube. Examine the gas evolved to see whether it is combustible.

2. Hydrogen from water (elective experiment).—Set up an apparatus as indicated in the diagram. (*A*) is a tube of hard glass filled with clean iron turnings or wire, (*b*) a flask containing water and provided with a safety

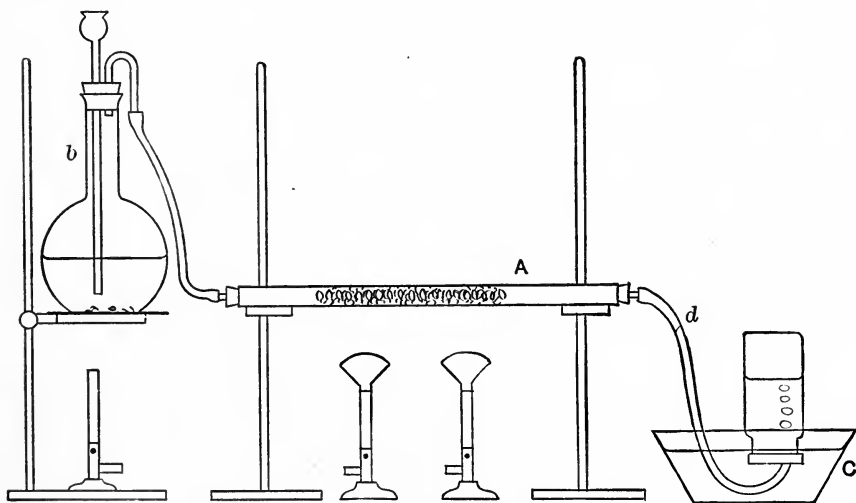


FIG. 7.

tube, (*C*) is a basin of water, and (*d*) a rubber tube. To prevent explosive boiling or "bumping," put into (*b*) *two* or *three* pieces of broken pipe stem or hard brick the size of grains of wheat. Heat until steam is passing freely

through (*d*), and until there is no longer a tendency for water to collect in (*A*). Then gradually heat the iron turnings to dull redness, and collect, by displacement of water, two or three bottles full of the escaping gas. Will the gas burn? Will it explode when mixed with air? What has become of the oxygen of the water?

3. Hydrogen from acids. — Put into a small test tube a few pieces of granulated zinc, Zn , cover with water, and add enough strong hydrochloric acid, HCl , to produce a rapid evolution of gas. Turn over the tube a larger tube, and let the action continue for a few moments. Remove slowly the larger tube, keeping the mouth of the tube down, and apply a flame. Repeat the same experiment, using sulphuric acid, H_2SO_4 , instead of hydrochloric acid. Again repeat, using iron wire or turnings instead of zinc. Can you make a statement which will cover all these cases?

4. Preparation of hydrogen. — Set up an apparatus as indicated in the cut. (*A*) is a flask or bottle, (*b*) a glass tube to which is attached the rubber tube (*c*), (*D*) is a funnel tube, (*E*) is a basin of water.

Into the flask put a small handful of granulated zinc, Zn , inclining the flask so as not to break it with the falling zinc.

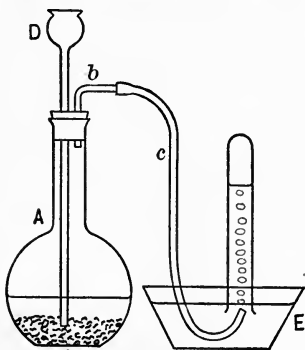


FIG. 8.

Cover the zinc with water, and after placing the stopper, add, through the funnel tube, enough hydrochloric or sulphuric acid to cause a brisk but not too rapid evolution of gas. Allow the hydrogen to bubble up into a test tube filled with water and held inverted in a basin of water, until it is filled. Apply a match. If it makes a whistling report, the hydrogen is not free from air, and *if ignited in a confined space would cause a dangerous explosion*. Successive test-tubefuls should be collected, until on applying a match no more noise is made, but only a slight snapping at the mouth of the tube, and the hydrogen burns only as the outer air comes to it. By testing in this way all danger is avoided and *in no other way can the danger be avoided*, no matter how many times the gas may have been *previously made* and no matter how long in each case the generator may have been in operation.

5. Density of hydrogen as compared to air.—(a) Collect a bottleful of hydrogen and set it with the mouth upward on the desk. After a moment apply a match. What has taken place?

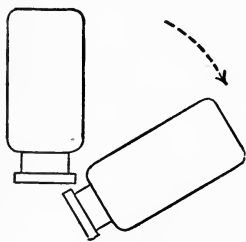


FIG. 9.

(b) Hold in one hand a bottle of air and in the other a bottle of hydrogen, each with the mouth down. Steadily bring the mouth of the hydrogen bottle below that of the air bottle and at the

same time incline it so that the hydrogen may be poured upward into the air bottle. Apply a match to each. What has taken place and why?

(c) Fill soap bubbles with hydrogen by dipping for a moment the end of the rubber tube into a strong soap solution and then letting the film be expanded enough by the gas to make it rise when shaken from the tube.

6. Chemical properties of hydrogen. — Being sure by the test given in (4), that the hydrogen escaping from the generator is free from air, put into the end of the rubber delivery tube the jet (*d*, Fig. 3). Before applying a flame be ready to note that when first ignited the hydrogen flame is colorless. It is only when the glass becomes hot that the flame is yellow from the sodium of the glass. Introduce into the hydrogen flame a piece of platinum wire. Kindle with it a match and a piece of charcoal. What is shown by these experiments?

CHLORINE

All experiments must be performed under the draught cupboard or "Hood."

7. Chlorine. — Warm a little crude hydrochloric acid, HCl , with a little potassium dichromate, $\text{K}_2\text{Cr}_2\text{O}_7$. Warm a little hydrochloric acid with a little potassium chlorate, KClO_3 , with a little lead peroxide, PbO_2 , with manganese dioxide, MnO_2 . Notice the evolution of the ill-smelling

chlorine in each case. The substances used with the hydrochloric acid are all such as easily give up oxygen. How do they set the chlorine free?

8. Preparation of chlorine.—Arrange a flask with a delivery tube, preferably of glass, and a funnel tube as indicated in Fig. 10. Into the flask put

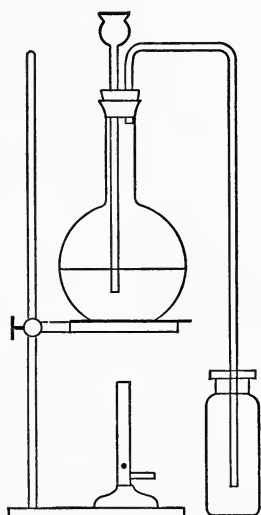


FIG. 10.

25 grammes of manganese dioxide, MnO_2 , and pour through the funnel tube 200 c.c. of crude hydrochloric acid. Warm with the burner and collect the evolved gas in wide-mouthed bottles by displacement of air. Is chlorine heavier or lighter than air? (If water comes over as steam, too much heat is being used, and more acid may be necessary.)

9. Properties of chlorine.—(a)

Into a bottle of chlorine sprinkle a little powdered antimony, Sb. What is formed?

(b) Heat one end of a thin strip of metallic copper and thrust it into a bottle of chlorine. What evidence is there that chemical change has taken place? What is formed?

(c) Warm a little turpentine, dip a strip of filter paper into it, and thrust it into a bottle of chlorine. What is the black smoke? Why is this set free?

(d) Put a very small piece of phosphorus, P, into a bottle of chlorine by means of a deflagrating spoon.* (*In handling phosphorus very great caution must be used; it must be kept under water as much as possible, and on no account must it be touched with the hands.*) (See 82.) What chemical characteristics of chlorine are illustrated by these experiments?

(e) **Bleaching action of chlorine.** — Into a bottle of dry chlorine put a piece of colored calico and cork tightly. Into another bottle of chlorine put a similar piece of calico which has been moistened. What difference do you notice in the two cases, and how is the difference explained? What can you say of the chemical conduct of chlorine?

10. Hydrogen chloride. — Into a flask fitted with a funnel tube and delivery tube put 40 grammes of common salt, NaCl, and pour through the funnel tube a somewhat diluted sulphuric acid made by pouring 50 c.c. of crude sulphuric acid into 10 c.c. of water. Heat on a piece of asbestos paper or a sand bath, letting the gas evolved bubble into 50 c.c. of water contained in a beaker. Why

* A strip of tin plate about 2 cm. wide and 20 cm. long makes a very convenient piece of apparatus. It may be used as a test-tube-holder by wrapping the middle around the tube and holding the ends. It may be used as a deflagrating spoon by bending it up for a centimetre at one end and bending about 10 cm. of the other end at right angles and in the opposite direction to the first bend. This will serve as a handle to keep the hand away from over the heat.

at first do the bubbles pass through? Then why do they not pass through? and, finally, why do they again pass? Notice the density currents in the water, bring the delivery tube near the surface of the water, and from the direction of flow of the currents judge whether the solution is more or less dense than water. (See 11.)

Remove the tube from the water and exhale through the mouth across the end of the tube. What causes the appearance of the cloud?

Hold a burning match in the current of the gas. Will it burn? Will the match burn in it?

Write a chemical equation to express the reaction between salt and sulphuric acid.

11. Hydrogen chloride as an acid. — (a) Into a portion of the solution in the beaker in (10) put a piece of blue litmus paper. Notice that the color can be made blue again by either sodium hydroxide, NaOH , potassium hydroxide, KOH , or ammonium hydroxide, NH_4OH .

(b) Dilute some of the solution quite largely and taste a little of it. Describe the taste. To 1 or 2 c.c. of a five per cent solution of caustic soda, NaOH , add the solution of hydrochloric acid until it is neutral, *i.e.* until it will not change blue litmus red or red litmus blue. Now taste of the solution. Describe the taste. What has been formed? Write the equation.

How much sodium chloride, by weight, can be made from 10 grammes of hydrogen chloride?

(c) Pour part of the solution on a few pieces of zinc in a test tube; apply a match to the mouth of the tube. What gas is evolved?

How much zinc, by weight, will be required to make one gramme of hydrogen?

OXYGEN

12. Preparation of oxygen. — Cork one end of a piece of hard glass tubing of about 6 mm. diameter and 15 cm. length. Put into the tube about a gramme of mercuric oxide, HgO . Connect to the open end of the tube a piece of rubber connector, lay the tube on a support, and heat with the burner provided with its wing top. (Have care not to burn the cork.) Collect over water one or two test-tubefuls of the gas and notice that a glowing coal at the end of a charred stick will burn so much more intensely in it than in air as to cause the stick to burst into flames.

From 15 grammes of mercuric oxide how many grammes of oxygen can be made? How many grammes of mercury? How many grammes of mercuric oxide are necessary to make 8 grammes of mercury?

13. Preparation of oxygen. — Fit a dry 15 cm. test tube with a cork and glass tube so bent that the test tube may be suspended by the bend from the ring

of the lamp stand. Fill the tube about one-fourth full of potassium chlorate, KClO_3 , and suspend as suggested.

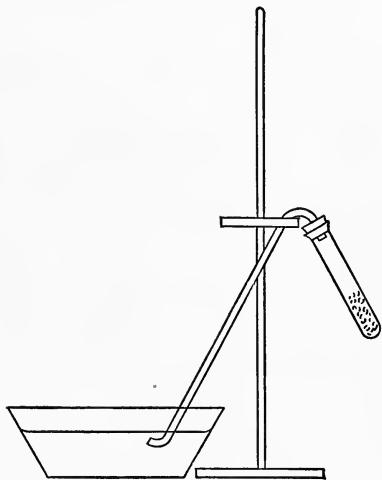


FIG. 11.

Heat with the burner, keeping the flame moving so as to avoid heating locally and melting the tube. The salt melts, boils, and a gas is given off. Collect over water a bottleful. Remove the delivery tube from the water and stop heating. After making an experiment with this portion of the gas a new portion may be obtained by again heating. (See 14.)

How much potassium chlorate will yield 1500 grammes of oxygen?

14. Chemical properties of oxygen. — (a) Kindle one end of a pine splinter, and when it is well burning blow out the flame and, while the charcoal at the end is still glowing, introduce it into a bottle of oxygen. Why is the combustion fiercer?

(b) Put some sulphur, S, on a deflagrating spoon and put it into a bottle of oxygen. (See note on Expt. 9, d.) Set it on fire and again lower it into the bottle of oxygen. Why the difference?

(c) Put into a deflagrating spoon a small piece of phosphorus, P. Warm in the flame until it catches fire and put it into a bottle of oxygen. (Caution! See 9, *d*.)

(d) Twist together a few strands of fine iron wire and thrust through a large cork. Wind a small shred of cloth around the end of the twist and dip it into molten sulphur. Kindle the sulphur and introduce into a bottle of oxygen. What common change is like that accompanying this brilliant display? Why is the sulphur necessary?

15. Ozone.—Into a beaker put a few small pieces of phosphorus which have been freshly cut from a larger piece. Nearly cover the phosphorus with water, and cover the beaker with a watch glass. After a short time lift the watch glass and notice the peculiar odor. Insert into the beaker a piece of filter paper moistened with starch paste (see 70) to which a little potassium iodide, KI, has been added. Notice the blue color due to the action on the starch of the iodine liberated by the ozone.

WATER

16. Occurrence of water.—(a) In organic matter: Heat in a dry test tube a little of any apparently dry organic matter, as wood, hair, leather, etc. Observe the deposition of moisture on the cooler parts of the tube.

(b) Water of crystallization: Heat in a dry test tube some crystals of copper sulphate, $\text{CuSO}_4 + 5\text{H}_2\text{O}$. Notice the change of color, loss of crystalline form, and deposition of moisture on the cooler parts of the tube. Repeat, using zinc sulphate, $\text{ZnSO}_4 + 7\text{H}_2\text{O}$, or calcium chloride crystals, $\text{CaCl}_2 + 6\text{H}_2\text{O}$.

What percentage by weight will be lost by each of these three salts when all of the water of crystallization is driven off?

17. Composition of water.—Arrange an apparatus for the generation of hydrogen (read Expt. 4). Place a jet in the delivery tube and kindle the issuing gas. Fill a beaker with cold water, and having carefully dried the bottom of the beaker hold it in the hydrogen flame. Notice the collection of the moisture on the bottom of the beaker, and express the formation of water by an equation. (In this experiment and the next the generator should not be allowed to heat up.)

18. Composition of water.—Attach to the hydrogen generator a hard glass tube in which a layer of copper oxide, CuO , has been placed. Test to be sure that all air has been driven from the apparatus, and then heat the copper oxide to dull redness while the hydrogen is passing over it. Notice the deposition of moisture in the cool part of the tube. Write an equation showing the reaction. (See page 54 for figure.)

From 100 grammes of hydrogen how many grammes of water will be formed? How much copper oxide will

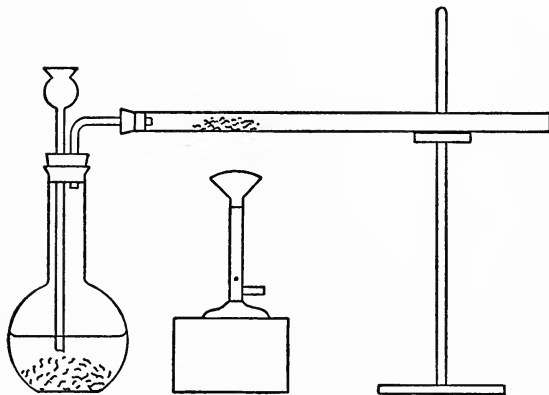


FIG. 12.

be necessary to furnish the oxygen contained in this amount of water?

19. Hydrogen peroxide.—(a) To a dilute solution of potassium dichromate, $K_2Cr_2O_7$, add a few drops of dilute sulphuric acid, H_2SO_4 , and a few drops of commercial hydrogen peroxide, H_2O_2 .

(b) Into a test tube containing a little water, put a pinch of lead peroxide, PbO_2 , or of red lead, Pb_3O_4 , and some hydrogen peroxide. Notice the change of color and the evolution of a gas. What is the gas? Test it. Write the equations.

(c) Into a test tube containing some hydrogen peroxide, put some hair or a feather and notice their loss of color.

(d) Dip a piece of paper saturated with starch paste and potassium iodide into a dilute solution of hydrogen peroxide. (Compare with experiment 15.)

SALTS OF THE OXYGEN ACIDS OF CHLORINE

20. Sodium hypochlorite. — Pass chlorine through a little water in a wash bottle, and then through a solution of 5 grammes of caustic soda, NaOH , in 100 c.c. of water until the solution is saturated. Put a piece of colored calico into the solution. Is it bleached? Then dip it into dilute sulphuric acid. To the solution add dilute sulphuric acid. What is given off? Write the reactions.

How many grammes of chlorine are required to make 10 grammes of sodium hypochlorite? How many grammes of chlorine will be set free by adding sulphuric acid to a solution made as above and containing 10 grammes of sodium hypochlorite?

21. Potassium chlorate. — (a) Put a little strong sulphuric acid on a very little potassium chlorate, KClO_3 . Notice the yellow color and the peculiar odor. (Do not approach the face to the mass.)

(b) Mix a little powdered potassium chlorate with a little powdered sugar on a paper. (*Do not powder them together or rub them in the mortar together. It is dangerous.*) Put the mixture on an iron dish or on a brick



in the hood and add a drop of concentrated sulphuric acid by means of a glass rod. Why is the mass kindled?

(c) Add to a little potassium chlorate some concentrated hydrochloric acid. Notice the evolution of chlorine. Why is chlorine liberated by the action of potassium chlorate? Compare experiment 7.

(d) Recall experiment 13. If necessary, repeat the first part.

22. Potassium perchlorate. — In an evaporating dish heat 25 grammes of potassium chlorate, stirring it with a glass rod. Occasionally take out with the rod a small amount and test it by bringing it into a little hydrochloric acid. Continue heating until chlorine is no longer evolved when tested in this way. What does this indicate? When cool, grind up the mass with a little water. Pour off the water and again treat with a little water, and pour off. Now heat with a little water, and, if necessary, add more water, until enough has been added to barely dissolve the salt when the solution is boiling. Set away to cool. The salt separating on cooling is potassium perchlorate, KClO_4 . What has been removed by the washing and crystallizing? (See Manipulation.) Filter off the salt, add to a little of it some hydrochloric acid. Add to another portion some concentrated sulphuric acid. Is it like potassium chlorate in these reactions?

Dry the salt and heat a part of it in a test tube, as in experiment 13.

How many grammes of potassium chlorate are required to yield 10 grammes of potassium chloride when the oxygen is all driven off? How much oxygen is produced at the same time?

How many grammes of potassium perchlorate are required to produce 10 grammes of potassium chloride when the oxygen is all driven off? How many grammes of oxygen are produced in this case? In what relation are the two amounts of oxygen? What law does this illustrate?

NITROGEN

23. Nitrogen from the air. — Cut a thin disk of cork from the end of an ordinary stopper. Float it on the water in a large basin. On the cork lay a small piece of phosphorus, P. (Caution! See 9, *d*.) Kindle the phosphorus and invert over it a bottle filled with air. Thrust a burning splinter into the gas remaining in the bottle. Will it support combustion? Will it burn?

One litre of hydrogen weighs .0896 gramme. How much does one litre of nitrogen weigh? What rule has been formulated indicating the connection between the molecular weight of a substance and its density in a gaseous form? What is the molecular weight of nitrogen?

24. Nitrogen by a chemical reaction. — To 50 c.c. of water in a small flask fitted with a delivery tube, add 10 grammes of potassium nitrite, KNO_2 , and 5 grammes of ammonium chloride, NH_4Cl . Heat the flask until action commences, then lower the flame and collect over water the gas evolved. Will it burn? Will it support combustion?

Write an equation showing by what chemical change the nitrogen has been made. From 5 grammes of ammonium chloride, how many grammes of nitrogen can be made?

25. Ammonia. — To a little solid ammonium chloride, NH_4Cl , on a watch glass, add a few drops of caustic soda, NaOH , solution. Notice the odor of ammonia. In a mortar grind together a little ammonium chloride and an equal quantity of quicklime, CaO . Notice the odor. Hold a piece of moistened red litmus paper in the vapor. What are the chemical changes in these experiments? How do ammonium chloride and ammonia differ in composition? What is taken away from ammonium chloride by the reaction?

26. Preparation and properties of ammonia. — Connect a 250 c.c. flask with two bottles arranged as shown in the figure. The arrangement of tubes is to keep the water from drawing back into (a), if the absorption by the water becomes more rapid than the evolution of am-



monia. Into (c) put 20 c.c. of distilled water. (B) is empty. Pulverize 30 grammes of quicklime and mix with

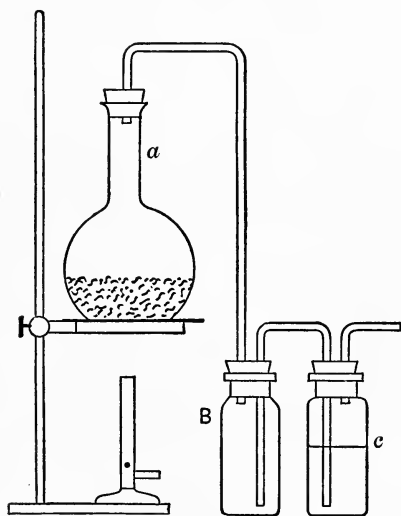


FIG. 13.

it 30 grammes of solid ammonium chloride. Pour the mixture into the flask and heat until the gas is no longer absorbed. How can this be known? Notice the density currents in (c). By observing the direction of their flow, determine which is the more dense, water or ammonia solution?

(b) Disconnect (B) from (a) and collect some of the gas by displacement of air in a dry test tube. Is it lighter or heavier than air?*

What is the density of ammonia as compared to hydrogen? How is this recorded in its formula? What is its density as compared to air? Which end of your tube should be up to collect ammonia?

(c) Put into a test tube a small piece of salt, sodium chloride, NaCl . Pour upon it a little strong sulphuric acid, H_2SO_4 . What gas is formed? (See 10.) Over

* Air is a mixture of 20.96 per cent of oxygen and 79.04 per cent nitrogen by volume. What is its average density as compared with hydrogen? Remember the result. It is an important number.

this so that the mouths of the tubes come together bring a test tube full of ammonia gas. Notice the dense cloud and the solid white deposit on the sides of the tube. What is it? Write an equation to express the reaction. Why does the tube become warm?

27. Dissociation of ammonium chloride (elective).—

(a) Dissolve a little ammonium chloride, NH_4Cl , in water. Test the solution with blue and red litmus paper. In a test tube heat a little ammonium chloride. When fumes are freely coming from the mouth of the tube, test their reaction with blue and red litmus paper. Are they acid or alkaline?

(b) Put a plug of long fibre asbestos in the middle of a hard glass tube 1 cm. in internal diameter, and 20 cm. long. Put a lump of ammonium chloride close to one side of the asbestos plug. In each end of the tube put two pieces of moist litmus paper, one blue and one red. Take the tube in the two hands and after lightly laying the thumbs over the ends of the tube heat the ammonium chloride and the asbestos plug. Which pieces of litmus paper change color? Exchange the pieces of litmus paper and heat again. Is there any change in color? Smell the two ends of the tube. Interpret. Why, when the salt was heated in the test tube, was there no acid or alkaline reaction?

28. Preparation of nitric acid.—Into a 50 c.c. glass stoppered retort put 25 grammes of sodium nitrate, NaNO_3 ,

and 15 grammes of concentrated sulphuric acid, H_2SO_4 . Heat the mixture and collect the nitric acid distilled over in a test tube kept cool by immersion in a basin of water. (See Fig. 6.) Preserve for use in 30, *b*. (Compare with 10.) How are the two experiments alike? Write the equation and make and solve a chemical problem based on this experiment.

29. Nitric acid as an acid.— Repeat experiment 11, using nitric instead of hydrochloric acid. Make a comparison between the two sets of reactions.

30. Nitric acid as an oxidizing agent.— (*a*) Pour some concentrated nitric acid diluted with an equal quantity of water on a little granulated tin, Sn, and warm it slightly. The white powder formed is principally an oxide of tin.

(*b*) In a test tube heat 1 or 2 c.c. of the nitric acid made in (28). Set fire to a pine splinter and when it is well charred at one end blow out the flame and thrust the glowing coal into the hot nitric acid. Notice the red fumes. What evidence have you that the coal is oxidized? (This experiment should be cautiously done to avoid contact of the acid with the fingers. It is best to hold the test tube over a large dish to avoid danger if it should break.)

31. Try the action of nitric acid on the metals copper, Cu, zinc, Zn, and iron, Fe. From the action of hydro-

chloric acid on zinc and on iron, and of sulphuric acid on the same metals, what might you expect when nitric acid acts on the metals? Is hydrogen easily oxidizable? (See 17 and 18.) Is there here present any substance from which oxygen is easily obtained? What becomes of the hydrogen? Write the equations.

32. Preparation of a nitrite.—Heat together in an iron dish 10 grammes of sodium nitrate, NaNO_3 , and 30 grammes of metallic lead, Pb, until the mass is dark brown and shows but little metallic lead. When cool add water, heat to boiling, and when the mass is disintegrated, filter. Test the filtrate in a small flask by adding to it some strong sulphuric acid. To another portion add a little potassium iodide, KI, and a few drops of dilute sulphuric acid. Test in the same way a solution of the original sodium nitrate. What differences are there? Explain.

33. The reduction of nitric acid to ammonia.—Into a 150 c.c. flask put a gramme of metallic aluminum in chips or clippings. Pour on this 25 c.c. of laboratory solution of sodium hydroxide, NaOH. Heat slightly. Notice evolution of hydrogen. Add four or five drops of concentrated nitric acid. After a few minutes, notice the odor of ammonia at the mouth of the flask. Test with red litmus paper. Bring near the mouth of the flask a glass rod moistened in strong hydrochloric acid.

(See 26, c.) What change has the hydrogen made in the nitric acid?

34. Nitric oxide and nitrogen peroxide.—Into a flask provided with a funnel tube and a delivery tube put a few grammes of metallic copper. (Best in the form of turnings.) Partially cover with water and add through the funnel tube 1 or 2 c.c. of concentrated nitric acid. Heat moderately and add more acid if necessary to produce a good evolution of gas. Collect a bottle-ful over water. What is its color? Cover the mouth of the bottle and set on the desk. Thrust a glowing coal into the gas. Will it burn or support combustion? What change in color is produced when air comes in contact with the gas? How is it explained? Write an equation showing by what chemical changes these gases have been made.

What is the density of nitric oxide as compared to hydrogen? What is the density of nitrogen peroxide? What will be the weight of a litre of each?

35. Nitrous oxide.—Into a 100 c.c. retort put 10 grammes of crystallized ammonium nitrate, NH_4NO_3 . Attach a delivery tube to the neck of the retort, heat carefully, and collect over water the gases evolved. Notice that the deposition of water in the neck of the retort continues all through the course of the experiment. Why? Is it due to moisture contained



in the original substance? Write the equation. (Compare with 24.)

Bring into a bottle of the gas a glowing splinter. (Compare with 34.) Bring into a bottle of the gas, on a deflagrating spoon, a little sulphur which is

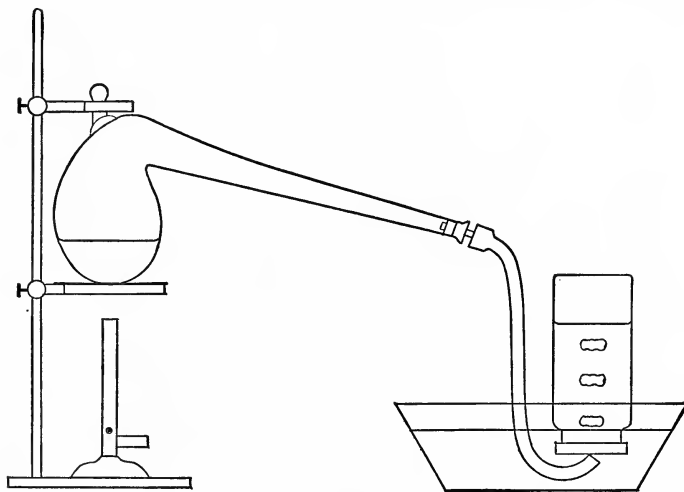


FIG. 14.

feebly burning. What is the result? Try it again, but with the sulphur burning strongly. How do you explain the difference in action?

From 85 grammes of ammonium nitrate, how many grammes of nitrous oxide will be formed? What is the density of the gas? How much will a litre weigh? How many litres can be produced from 85 grammes of ammonium nitrate? If all the oxygen in 5 litres of nitrous oxide is taken away, how many litres of nitrogen will be left?



CARBON

36. Into a dilute solution of indigo in a test tube, put a small quantity of animal charcoal, and after thoroughly shaking pour on a filter. What change has taken place in the color of the solution? Try the same experiment, using a stronger solution of indigo. If the solution is not decolorized, pour it again upon the charcoal and boil. Again filter and observe color.

37. Carbon dioxide.—Arrange an apparatus as indicated in the cut. (*A*) is a flask filled with air. (*B*) is a hard glass tube containing fragments of charcoal.

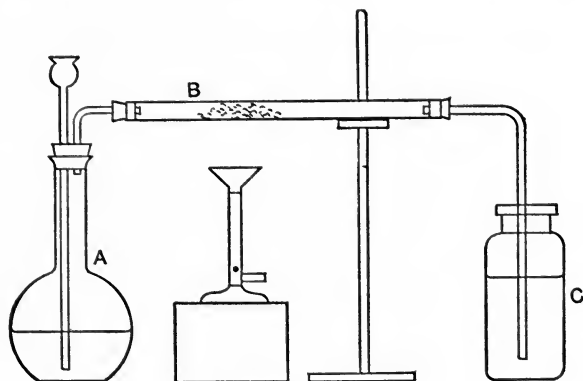


FIG. 15.

(*C*) is a bottle containing freshly filtered lime water. Pour water through the funnel tube to force air through the apparatus. What change takes place in the lime water? Now heat the charcoal to redness, and again

pour water into (A). Is there any evidence of a change going on in (B)? Is the lime water changed in appearance? Why?

38. Carbon dioxide.—Arrange an apparatus as in Fig. 8. Put into the flask a handful of coarsely broken marble and enough water to cover it. Pour through the funnel tube enough crude hydrochloric acid to cause a brisk evolution of gas. Collect a bottleful by displacement of water. Thrust a burning splinter into it. What takes place? Collect two more bottlefuls in the same way. Hold one of them with the mouth up, and the other with the mouth down. In a short time put a burning splinter into each. From what you see, what can you say as to the relative density of carbon dioxide and air?

Fill a bottle with the gas by displacement of the air. Pour it into another bottle as you would pour water, and detect its presence in the second bottle by the action on a burning stick. Is this great density of carbon dioxide recorded in any way in its formula? What is the theoretical density as compared to hydrogen? As compared to air? (See note to 26.)

39. Pass carbon dioxide into freshly filtered lime water.

40. Pass air from the lungs into clear lime water. (Compare 37.)

41. Pass carbon dioxide from the generator into clear lime water and continue to pass it until the precipitate first formed redissolves. Now boil the clear liquid.

42. (a) Fill a small test tube with carbon dioxide, cork the tube and invert it in a small beaker filled with caustic soda solution, NaOH. Remove the cork with a pair of forceps. How do you explain what takes place?

(b) Pass the gas into the caustic soda solution until it is no longer absorbed. Add to some of this solution an acid. Why in (39) is the precipitate formed and not in (42)?

43. Bring together any carbonate and any acid. Bring together any other carbonate and any other acid. What is given off?

44. Pass carbon dioxide into 5 c.c. of cold distilled water for a few minutes. Test the liquid with blue litmus paper. Add to a portion of the liquid some clear lime water. Has the gas imparted to the water any of its properties? What is supposed to be contained in the liquid? Boil and test again.

45. Carbon monoxide (hood). — Arrange an apparatus as shown in the cut. Into the 500 c.c. flask put 10 grammes of oxalic acid, $\text{H}_2\text{C}_2\text{O}_4$, and 50 c.c. of strong sulphuric acid. The two wash bottles contain a strong

solution of caustic soda, NaOH . Heat carefully and collect through (a) the escaping gas over water. Kindle the gas in one of the collecting bottles. What is formed by the combustion? After the gas has passed for some time, pour out into a test tube part of the contents of the wash bottle nearer the generator, and add to it an acid. Put a burning match into the top of the

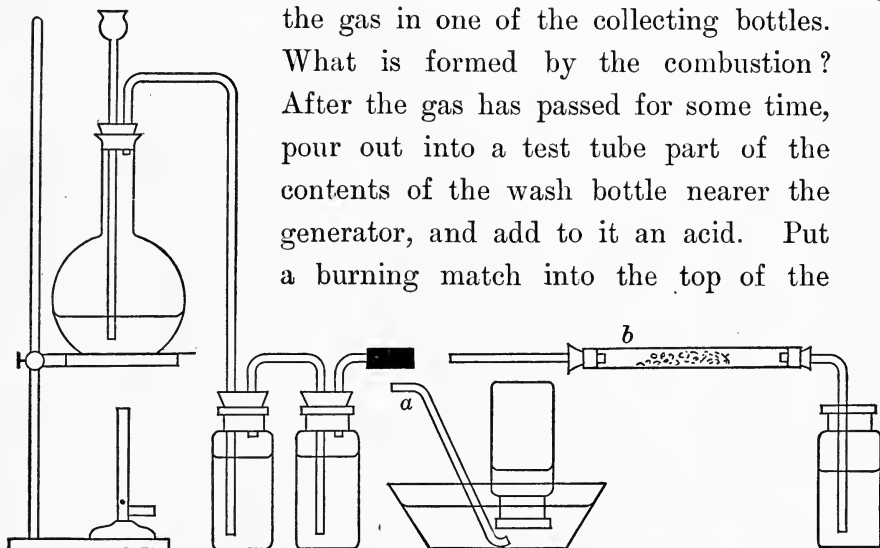


FIG. 16.

tube. Does it burn like carbon monoxide? (Compare 42, b.) What reaction has taken place in the flask?

46. Reducing power of carbon monoxide. — Connect with the apparatus described in (45) a hard glass tube (b) containing a little copper oxide, CuO . Pass the gas escaping from (b) into clear lime water. (If a cloudiness is produced, to what is it due? How can the cause be removed?) If no cloudiness is produced, heat the copper oxide, observe the change in color as the black oxide is changed to metallic copper. Also observe the cloudiness now produced by passing the

gas into clear lime water. Explain the phenomena, and write the equation to show how the reduction has been effected.

How many litres of carbon monoxide are required to reduce 50 grammes of copper oxide? How many litres of carbon dioxide will be produced? If the oxygen taken from the copper oxide were in a free state, how many litres would it make? Compare the results of these three problems. Can you solve the last two problems without calculation by inspecting the equation.

Cyanogen compounds (elective).—In working with potassium cyanide or other cyanogen compounds the greatest caution must be used, as they are for the most part *deadly poisons*. Do not add acids to them except under a hood. Do not touch them with the hands.

47. Double cyanides (elective).—Make a solution of potassium cyanide, KCN. Add to it a little solution of silver nitrate, AgNO_3 , noticing that it produces a precipitate which dissolves in an excess of the cyanide. Add another portion in the same way to a solution of nickel sulphate, NiSO_4 .

48. Potassium cyanate (elective).—Heat in a porcelain crucible 1 gramme of potassium cyanide and 3 grammes of litharge, PbO , until all is fused. Pour out



and notice the metallic lead. What has become of the oxygen of the litharge?

49. Potassium sulpho-cyanate (elective). — Repeat experiment 48, using lead sulphide, PbS , instead of litharge. Dissolve the product in water, add a few drops of hydrochloric acid, and add then a little solution of ferric chloride, FeCl_3 .

50. Sodium ferro-cyanide (elective). — To a solution of caustic soda in a small flask add a little solution of ferrous sulphate, FeSO_4 , and then a little potassium cyanide. Boil for a few minutes, dilute and filter. Acidify the solution with hydrochloric acid and add ferric chloride solution.

Write equations showing the reactions, and make a statement of the properties of the cyanogen compounds.

SODIUM AND POTASSIUM (*base-forming elements*)

51. Sodium hydroxide. — To 25 c.c. of water in an evaporating dish add, one at a time, three or four pieces of sodium the size of a pea. Have care to avoid the spattering at the end of the action of each piece. What causes it? Dilute a little of the solution thus made and taste it. Dip the finger in the solution, and notice the soapy feeling when the finger and thumb are rubbed together. Dip a piece of red litmus paper in the solution. Clean a piece of plati-

num wire by dipping it into hydrochloric acid, and heating it in the flame until it no longer imparts a color to the flame. Dip the clean wire into the solution and heat again. Notice the color of the flame.

52. Sodium hydroxide. — Dissolve 10 grammes of barium hydroxide, $\text{Ba}(\text{OH})_2$, in 100 c.c. of hot water. Add to the hot solution a solution of sodium sulphate until no more precipitate is formed, but add no more than is needed for complete precipitation. Filter, dilute, and taste. Rub between the finger and thumb. Test with litmus paper. Note the flame color. In a small iron or silver dish evaporate to dryness.

How much sodium sulphate, $\text{Na}_2\text{SO}_4 + 10 \text{H}_2\text{O}$, will be needed to exactly precipitate 10 grammes of barium hydroxide, $\text{Ba}(\text{OH})_2 + 8 \text{H}_2\text{O}$?

53. Preparation of a salt. Neutralization. — Dissolve 2 or 3 grammes of sodium hydroxide in 10 c.c. of water, and add to the solution with stirring barely enough hydrochloric acid to take from it the property of turning red litmus paper blue. Evaporate to complete dryness. Taste the residue. Add to a part of it some concentrated sulphuric acid. What is given off? What color does the salt impart to the flame?

54. Preparation of a salt. Double decomposition. — Dissolve 5 grammes of barium chloride, $\text{BaCl}_2 + 2 \text{H}_2\text{O}$, in

50 c.c. of hot water, and while the solution is still hot add a solution of sodium sulphate in small portions until no more precipitate is formed. *Avoid an excess.* Filter. Taste the filtrate.

Write equations for the reactions involved in experiments 53, 54, 10, and 28. What do you mean by a basic element? What is a salt? What general methods of making salts are illustrated by the experiments referred to? (Compare also 20, 21, 22.)

55. Potassium hydroxide. — Repeat with potassium, experiment 51. (Caution.)

56. Thoroughly clean a platinum wire and observe the color given to the flame after dipping it into potassium chloride. Observe the color through a blue glass or an "indigo prism." Add to the solution a little common salt and observe the flame color with and without the blue solution.

57. To a cold solution of potassium chloride add a cold solution of acid sodium tartrate, $\text{NaH}(\text{C}_4\text{H}_4\text{O}_6)$. If a precipitate fails to form, rub the inside of the test tube below the surface of the liquid with a glass rod. The precipitate formed is acid potassium tartrate, $\text{KH}(\text{C}_4\text{H}_4\text{O}_6)$.

What difference is there between the acid tartrates of potassium and sodium?

CALCIUM, STRONTIUM, AND BARIUM *

58. Barium.—Make a saturated solution of 10 grammes of barium hydroxide, $\text{Ba}(\text{OH})_2$. Filter. Force air from the lungs through a portion of the clear solution. Neutralize a portion of it with dilute nitric acid. What is now contained in the solution?

59. To separate portions of this solution, add solution of sodium sulphate, Na_2SO_4 , of sulphuric acid, H_2SO_4 , of ammonium carbonate, $(\text{NH}_4)_2\text{CO}_3$, of potassium chromate, K_2CrO_4 , and of sodium phosphate, Na_2HPO_4 .

60. Dip a clean platinum wire in the solution and hold it in the colorless flame of the Bunsen burner.

61. Strontium.—With a solution of strontium nitrate, $\text{Sr}(\text{NO}_3)_2$, or chloride, SrCl_2 , make the same tests as indicated for barium.

62. Add to concentrated solution of the strontium salt part of the barium hydroxide solution prepared in (58).

63. Calcium.—With a solution of calcium nitrate or chloride make the same tests as for strontium.

64. Prepare lime water by slaking a piece of quicklime, CaO , mixing with a large quantity of water and

* In making experiments with these elements distilled water and chemically pure (C.P.) acids should be used.

filtering. Force air through part of it from the lungs.

What similarities and differences in the three elements? Which hydroxide is the most soluble?

CHLORINE GROUP. HALOGENS

All experiments should be done under a hood.

65. Bromine. — Into a small retort arranged as in Fig. 6 put 1 gramme of potassium bromide, KBr , 2 grammes of manganese dioxide, MnO_2 , and 10 c.c. of strong sulphuric acid. Heat the retort and collect the distillate.

To the distillate add 10 c.c. of water, and stir. Notice the color of the solution. To a portion of this aqueous solution add a drop of chloroform, CHCl_3 , and shake. Note the appearance on standing, and explain.

66. Dissolve a small piece of potassium bromide in water, add a few drops of chlorine water. Notice the color change. Add a drop of chloroform, shake and let stand. Notice color of the chloroform and of the water. What change has the chlorine produced?

67. Hydrobromic acid. — Pour a few drops of strong sulphuric acid on a few pieces of potassium bromide in a test tube. Breathe across the mouth of the tube.

To what is the cloud due? Hold a piece of moistened blue litmus paper in the mouth of the tube. To what is the brown color of the vapor due? Examine a crystal of potassium bromide. What shape has it?

Make the same experiments with potassium chloride and compare. Notice the shape of the crystals.

68. Iodine. — Put a few small crystals of iodine in a test tube and warm them until they are volatilized. What do you see? What is the dark substance in the upper part of the tube?

69. Solubility of iodine. — Dissolve a little iodine in each of the following solvents: (1) water, (2) alcohol, (3) solution of potassium iodide, and (4) chloroform, or carbon disulphide, CS_2 . If the solutions are too concentrated to allow their color to be seen, dilute them.

70. Into a 15 cm. test tube, two-thirds full of water, put a piece of starch as large as a grain of wheat. Shake until the lump of starch is disintegrated and then boil.

(a) Cool the solution, and add to a part of it a dilute solution of iodine. Dilute the iodine solution and try it again. Heat the colored solution thus produced and then cool. What phenomena are observed?

(b) To another portion of the starch paste add a little potassium iodide solution. Then add a little

chlorine water. Then add much chlorine water. How do you explain the phenomena?

How many litres of gaseous chlorine will be necessary to liberate 10 grammes of iodine?

71. Preparation of iodine.—In a test tube put a few crystals of potassium iodide with twice the bulk of manganese dioxide, MnO_2 , and enough sulphuric acid to moisten the whole. Heat gently, keeping the upper part of the test tube as cool as possible. (Compare 8 and 66.)

72. Hydriodic acid.—Pour a few drops of strong sulphuric acid on a small piece of potassium iodide in a test tube. What do you see and smell? (Compare 68 and 10.) Explain why colored gases are evolved with the iodide and bromide, but not with the chloride.

73. Fill out the following table:

	Cl.	Br.	I.
Element, how made? (Use symbols only of reagents used.)			
Gas, liquid, or solid?			
Color in gaseous form?			
Solubility in water?			
Stability of acid?			
Shape of crystals of potassium salt?			

FLUORINE

Fluorine is classed with the halogens. According to analogy, how might fluorine be made? What difficulty is there in making it?

74. Warm a piece of plain glass and coat it with paraffin. When cold, scratch through the paraffin the formula, SiF_4 , so as to expose the glass. Mix together in a lead dish powdered fluor spar, CaF_2 , and strong sulphuric acid, to make a thin paste. Put over this the paraffined glass and let it stand over night. Warm the glass and wipe off the paraffin. What has become of the silicon of the glass in the lines of the letters?

Fluorine. — (See also 103.)

SULPHUR

75. Into a 15 cm. test tube put 2 or 3 grammes of sulphur, and heat. Note, as the sulphur grows hotter: (1) fusion, (2) thickening, (3) second fusion, (4) boiling, (5) color of vapor, (6) condensation of liquid sulphur on sides of the tube.

76. Amorphous sulphur. — When the sulphur boils, pour it into a dish of cool water and examine the sulphur.



77. Rhombic sulphur. — Dissolve some powdered roll sulphur in carbon disulphide, CS_2 , and set it where the solvent will slowly evaporate. Examine the crystals.

78. Monoclinic sulphur. — Heat in a four inch Hessian crucible 40 or 50 grammes of sulphur until it is all melted, but not overheated. Cool, and when a crust forms over the top, break two holes in the crust, and through one of them pour off the sulphur still molten. When the crucible is cool, carefully break out a portion of the crust and examine the crystals formed.

How do the three forms of sulphur differ? What is the relation which exists between them called?

79. Heat 2 or 3 grammes of sulphur in a test tube to boiling, and thrust into the vapor a thin strip of metallic copper. Why does the copper become red hot? Notice the change in the character of the copper. What other cases have you had similar to this?

80. Hydrogen sulphide (hood). — In the generating flask (Fig. 8), put a handful of coarsely broken, ferrous sulphide, FeS . Cover it with water, and add through the funnel tube enough sulphuric or hydrochloric acid to start a good evolution of gas. Pass the gas through a little water, contained in a wash bottle, and collect a bottleful by displacement of water.

(a) Apply a flame. What takes place? What, from

your own observation, is one of the products of combustion? Write the equation.

From 8 litres of hydrogen sulphide how many litres of sulphur dioxide can be made? How many litres of oxygen are necessary for the complete combustion of 8 litres of hydrogen sulphide?

(b) Pass the washed gas for a few minutes into a little water in a bottle. After blowing away the gas above the water, shake and smell of the water. What does this odor indicate? How is it that you can collect the gas over water?

(c) **Sulphides.** — Pass the gas through dilute solutions of the salts; lead nitrate, $\text{Pb}(\text{NO}_3)_2$, zinc sulphate, ZnSO_4 , and arsenic trioxide, As_2O_3 , using hydrochloric acid as a solvent in the last case.

(d) Hold a piece of clean lead or a silver coin in the stream of gas.

81. Sulphur dioxide. — Into a generating flask provided with a funnel tube, put 25 grammes of metallic copper, preferably in the form of turnings. Add 50 c.c. of strong sulphuric acid and heat on a piece of wire gauze or asbestos paper. Collect by displacement of air a bottleful of the gas evolved. What record of its density is contained in its formula? Is it heavier or lighter than air?

(a) Introduce into a bottle of the gas a flower, some grass, or a piece of clean moist sponge, and continue

to pass the gas into the bottle. What change is there in the color?

(b) Collect another bottleful of the gas and invert it in a basin of water. Why does the water rise in the bottle?

(c) Pass the gas into about 20 c.c. of water. Is the gas readily absorbed? Test it with litmus paper. When it is saturated, add about a gramme of solid caustic soda, and when this is dissolved pass the gas again. Is the gas again absorbed? Why? Continue to pass the gas until the solution is saturated. Add to a portion of it a few drops of concentrated sulphuric acid. What is given off? How do you know?

(d) Pass sulphur dioxide into a dilute solution of potassium permanganate, about 10 c.c., until the color disappears. To what property of the sulphur dioxide is the change in color due?

82. Sulphuric acid.—(a) Into 10 c.c. of water in a test tube, pour 5 c.c. of strong sulphuric acid. Notice the heat produced.*

(b) Dip a match stick into strong sulphuric acid.

(c) Put a little sugar into strong sulphuric acid. (d) Dip a filter paper into dilute sulphuric acid and dry it. How do you account for the color changes?

* In diluting sulphuric acid always pour the acid into the water, and not the reverse. The heat generated by the contact of the water with the large amount of acid, may be great enough to cause the acid to be spattered about.

83. Formation of sulphuric acid (elective; hood).— Arrange an apparatus as shown in the figure. Connect with (a) an apparatus for making sulphur dioxide (81).

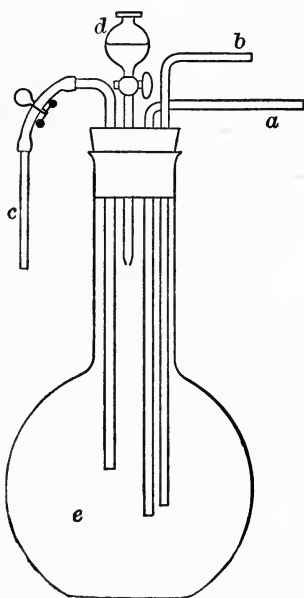


FIG. 17.

With (b) connect an apparatus for making nitric oxide (34). Pass both gases into the flask, and notice the deposit of crystals on the sides of the flask. When the gases in (e) become colorless, blow into it through (c). When they are again colorless, drop in two or three drops of water from (d), and notice the disappearance of crystals and formation of colored gases. Repeat these operations for fifteen or twenty minutes, then disconnect the apparatus and pour out the contents of the flask into an

evaporating dish, and evaporate until white fumes appear. What are they? Put a match stick into the liquid remaining in the dish. Explain the phenomena, and write equations showing the reactions which have taken place.

84. Sodium thiosulphate, "Hypo" $\text{Na}_2\text{S}_2\text{O}_3$. — Dissolve 15 grammes of caustic soda in 50 c.c. of water. Divide the solution into two equal parts. Saturate one-

half with sulphur dioxide, and then add the other half. What are the reactions?

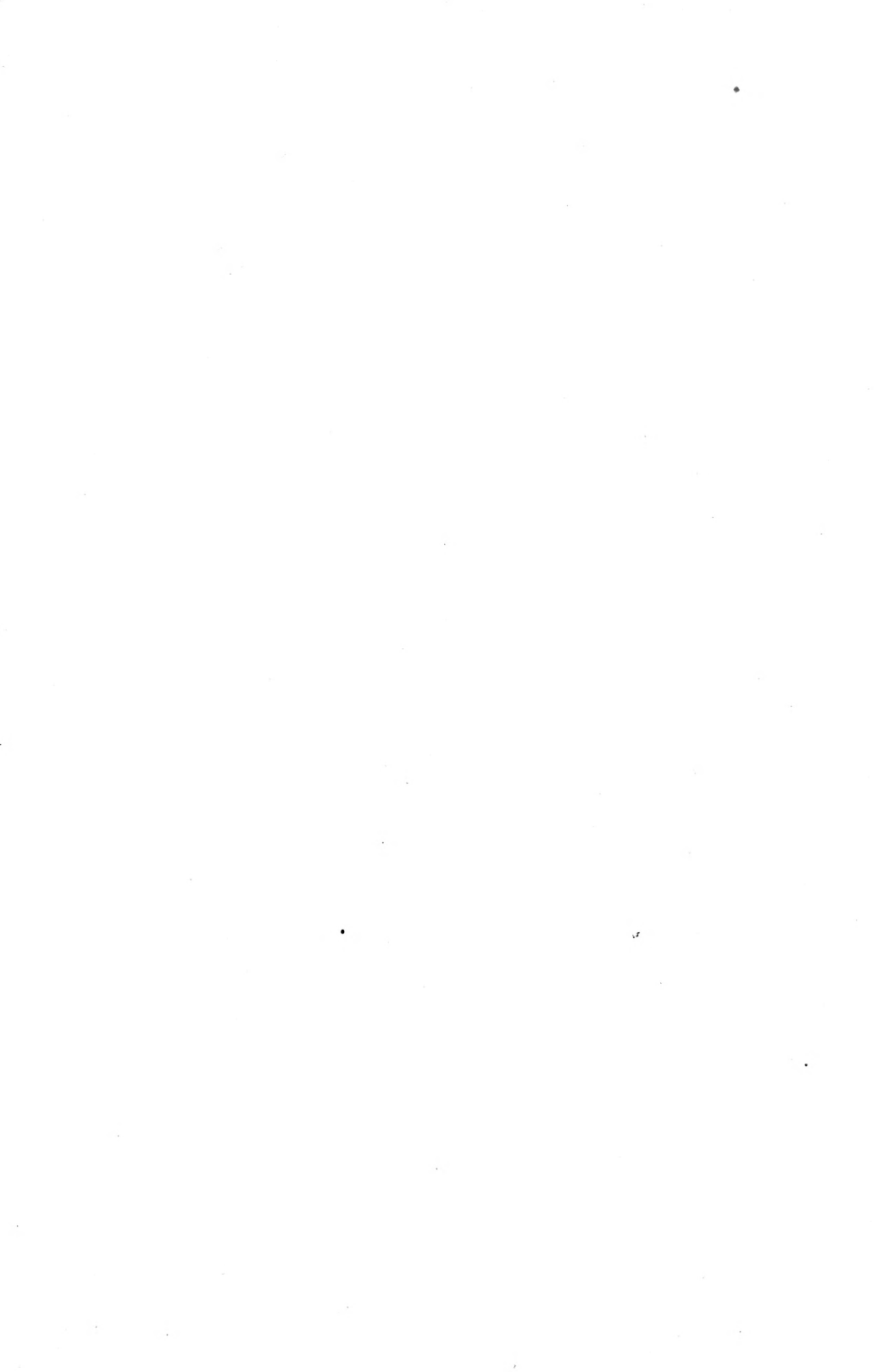
To the solution so formed add 5 grammes of flowers of sulphur and boil for twenty minutes, or until all of the sulphur is dissolved. Filter and evaporate until a solid begins to separate at the edges. Set away to crystallize. Lay the crystals formed on a piece of filter paper to dry.

To 1 c.c. of silver nitrate solution, AgNO_3 , add a little ammonium chloride solution, and then a solution of some of the salt made above. Describe the phenomena and explain, using equations.

PHOSPHORUS FAMILY

In working with phosphorus, the greatest caution must be used. — It must be kept and carried under water. It must on no account be touched with the fingers, even if the desk or floor seems to be in danger of being set on fire. If a piece of phosphorus catches fire, *let it burn out*, watching it to see that it sets nothing else on fire. It is better that it should burn while it is being watched. It may catch fire when no one is near, after the water with which it was quenched has dried off.

85. Phosphorus. — Put a piece of phosphorus the size of a grain of wheat into a test tube and add a few drops



of carbon disulphide, CS_2 . Under the hood, pour the solution thus formed on a piece of filter paper and watch until the solvent is evaporated. Caution!

86. Red phosphorus. — In a test tube 10 mm. in diameter and 10 cm. long, put a piece of phosphorus the size of a small pea. Cork very tightly. Now, with a test-tube holder, hold the tube above a burner with a small flame at such a distance that the phosphorus boils slightly. Keep it at that temperature until there is a very decided change in color. When the tube is cold, uncork it and fill it one-fourth full of carbon disulphide. After it has stood for a few minutes, pour it out on a filter paper. Is it all dissolved? Again treat with carbon disulphide. What is the residue?

87. Put a few grains of iodine in a test tube, add a little water, and sprinkle in red phosphorus a little at a time until the iodine disappears.



Notice the evolution of hydriodic acid.

88. Phosphorus trichloride (elective; hood.) — Fill a dry 150 c.c. retort one-fourth full of clean dry sand. Arrange as in the cut. The tube (a) is fitted through the cork in the tubulus loosely enough to permit of its

being pushed down or drawn up easily and at the same time tightly enough to keep its place. Pass carbon dioxide into the apparatus through the side neck of the *dry* 75 c.c. distilling flask used as a receiver. Continue passing it while filling in the phosphorus and until the chlorine apparatus is attached. Set up an apparatus for making

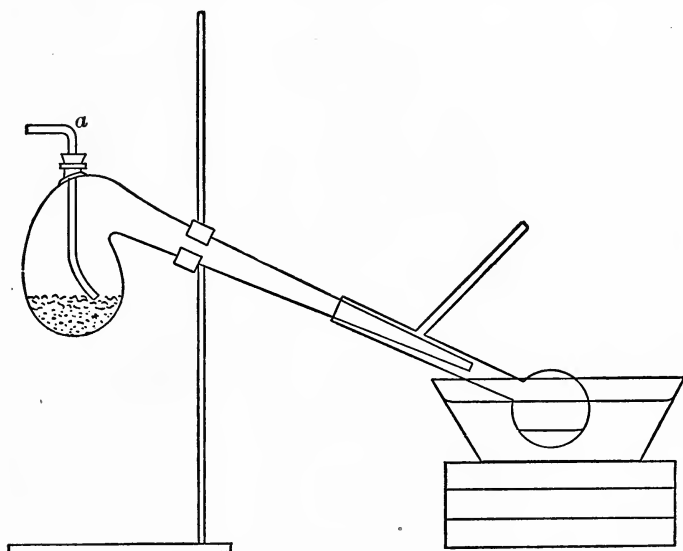


FIG. 18.

chlorine, using enough manganese dioxide to make three times the calculated amount of chlorine and other things in proportion. Pass the gas, to dry it, through a bottle of concentrated sulphuric acid. Break up phosphorus under water into pieces small enough to pass through the tubulus of the retort. Counterbalance a beaker with water in it. Then add a 10 gramme weight to the oppo-



site pan of the balance and put pieces of phosphorus into the beaker of water until equilibrium is restored. Take

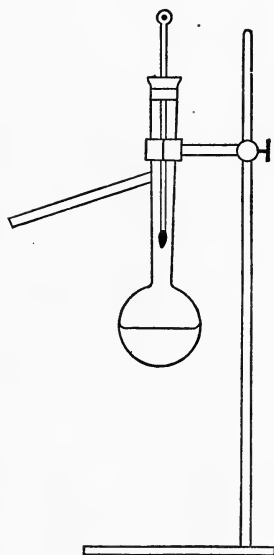


FIG. 19.

out the pieces of phosphorus, one by one, and dry them by wrapping them in filter paper for a moment. Quickly drop them into the retort which is overflowing with carbon dioxide. When all of the phosphorus is in the retort, connect with the chlorine apparatus. The phosphorus will soon catch fire in the chlorine. If a crystalline white deposit forms in the top of the retort, it is due to the formation of phosphorus pentachloride. In that case, push the delivery tube down. If a deposit of red phosphorus is formed, pull the delivery tube up. By adjusting this tube in depth and by rotating it, the greater part of the phosphorus may be changed into the liquid trichloride, which condenses in the neck of the retort and collects in the distilling flask. Redistil from the distilling flask, using the thermometer to note the boiling point. The temperature should vary but one or two degrees from 74°C . The whole operation must be conducted in a draught cupboard with a good draught.

Put a little of the phosphorus trichloride into a little water. What is formed?

Submit your calculations of the amounts to be used to an instructor before beginning to work. Why is it necessary to have all the apparatus dry?

89. Phosphine (hood). — *Read your text book.* Provide a 50 c.c. flask with a stopper and delivery tube.

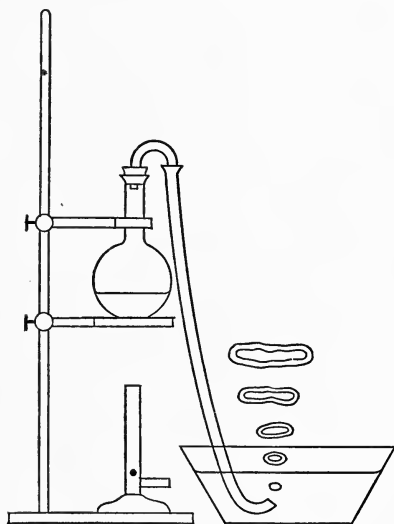


FIG. 20.

Fill the flask half full of concentrated solution of caustic soda. Drop in a piece of phosphorus the size of a pea. Support the flask on a piece of asbestos paper so that it can be heated. Pour into the flask one-half of a cubic centimetre of ether, $C_4H_{10}O$. On beginning to heat, the ether will be volatilized and expel all of the air before the reaction sets in. Place the stopper,

and, carefully keeping the delivery tube below the surface of the water in a large dish, heat nearly to the boiling point until enough bubbles have escaped to show the properties of the gas. Then heat more strongly until the delivery tube is hot with the escaping steam. Withdraw the lamp, close the hood, and wait until the water has drawn back into the flask before removing the stopper.

90. Phosphates.—To separate portions of a solution of “microcosmic salt,” $\text{NaNH}_4\text{HPO}_4$, add solution of calcium chloride, CaCl_2 , of ferric chloride, FeCl_3 , of lead nitrate, $\text{Pb}(\text{NO}_3)_2$, and of silver nitrate, AgNO_3 .

91. Metaphosphates.—Make a loop in the end of a platinum wire, by bending it around the point of a pencil, so as to make a loop about 3 mm. in diameter. (Do not twist the end around the main part of the wire as it is then harder to clean.) Heat the loop and dip it into crystallized microcosmic salt and heat again. When it is boiling in the flame, remove it and smell of it. What do you detect? Continue heating until there is no more bubbling. When the mass is cool, remove it by straightening out the loop and preserve it. Repeat the operation three or four times. Grind up the beads with a little water in a mortar. To separate portions of this solution, add the reagents used in the last experiment, and make a tabular comparison of the results obtained. Write equations and explain.

ARSENIC

92. Arsine (hood; caution!).—Arrange an apparatus as indicated in the cut. (*A*) is an apparatus for the generation of hydrogen with zinc and hydrochloric acid. (*B*) is a tube filled with granulated anhydrous calcium chloride, CaCl_2 , to dry the gas. (*C*) is a hard glass tube of 20 cm. in length and 5–7 mm. in diameter,

contracted at one end to a jet. Start a not too rapid evolution of hydrogen, and then add a little arsenic trioxide dissolved in hydrochloric acid. Why is the evolution of gas more rapid? When all of the air is expelled (see 4), ignite the gas at the jet and hold

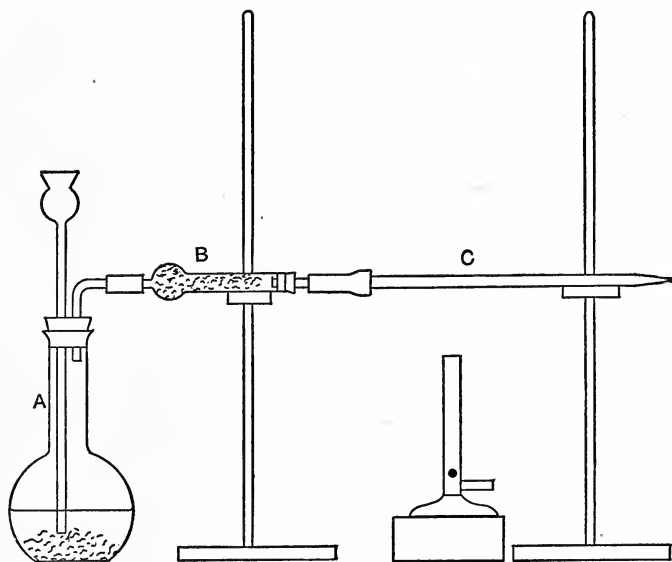


FIG. 21.

in the flame the porcelain pestle of a mortar. What are the dark spots? What are the white fumes beyond the flame? With a burner, heat the hard glass tube and notice the dark deposit on the cool part of the tube. Heat the deposit carefully. What becomes of it? Treat the spots on the porcelain and the deposit in the tube with a solution of sodium hypochlorite, NaClO . (See 20.) Why are they dissolved?

93. Arsenious acid and its salts.—Stir with water a few decigrammes of arsenic trioxide. Test the solution with blue litmus paper. What substance is supposed to give the reaction? How is the substance formed? (Compare 44 and 81, *b*, *c*.) To what class of substances do carbon dioxide, sulphur dioxide, and arsenic trioxide belong?

Heat the arsenic trioxide and water. Add drop by drop solution of caustic soda until *nearly* all is dissolved. Do not use too much soda. To portions of the solution made in this way, add solutions of copper sulphate, CuSO_4 , and of silver nitrate, AgNO_3 , and of magnesium sulphate, MgSO_4 . Keep the precipitates to compare with those made in (94).

94. Arsenic acid and its salts.—In a test tube, heat a few decigrammes of arsenic trioxide, with concentrated nitric acid, until no more red fumes are given off. Dilute, add an excess of ammonia water, and evaporate to dryness. What is the residue? Dissolve the residue in water and test it with the reagents used in (93), and compare the results of the two sets of tests.

ANTIMONY

95. Stibine.—Repeat (92), using instead of arsenic trioxide a solution in water of tartar emetic, $\text{SbOK}(\text{C}_4\text{H}_4\text{O}_6)$. What are the differences in the two cases?



96. Heat a little metallic antimony with concentrated nitric acid. Will the product dissolve in caustic soda? (Compare 94.)

97. Dissolve a little antimony trioxide, Sb_2O_3 , in concentrated hydrochloric acid. Dilute largely with water. What is formed? (See 98.)

98. Pass hydrogen sulphide into the emulsion formed by adding water to the antimony solution.

BISMUTH

99. Heat a little bismuth with concentrated nitric acid. (Compare 94 and 96.) Dilute very largely. Add a few drops of hydrochloric acid. (Compare 97.) Pass hydrogen sulphide. (Compare 98.)

BORON

100. **Borax as a flux.** — With borax, $\text{Na}_2\text{B}_4\text{O}_7$, make a bead as described in (91). While the borax bead is hot, dip it into copper oxide, CuO , or any copper compound in such a way as to take up a *very little* of the substance. Heat again in the flame, and notice the color of the bead when cold. Clean the wire, make a new bead and color with a little of some iron compound. Of what practical, everyday use is this property of borax?

101. Boric acid. — Dissolve 10 grammes of borax in 50 c.c. of boiling water. Add 5 c.c. of concentrated sulphuric acid. On cooling, boric acid will crystallize out. Filter off and wash on the filter with a little water. Dissolve in as little hot water as possible and let cool. Why? Again filter off, and dry by pressing between filter papers. Heat some of it on a loop of platinum wire.

102. Ethyl borate. — Cover a little powdered borax, in an evaporating dish, with alcohol and kindle the alcohol. Now blow out the flame, and add a few drops of concentrated sulphuric acid and kindle again. How do you explain the difference in the two cases?

SILICON

103. Alkali silicates. — Mix together one part of finely ground sand and five parts by bulk of each of sodium carbonate, Na_2CO_3 , and potassium carbonate, K_2CO_3 . Put about 1 c.c. in depth of the mixture in a small iron crucible, and heat until the mass is in a state of calm fusion. When cold, boil with 50 c.c. of water until the mass is disintegrated. Filter. To a portion, add concentrated hydrochloric acid drop by drop until a jelly-like precipitate is formed. If this fails to appear, evaporate the residue to half its bulk and again add hydrochloric acid. What is the precipi-

tate? Add excess of acid and evaporate to dryness. Dissolve as much as possible in hydrochloric acid. What is the residue?

Pass carbon dioxide into another portion of the solution of silicates. Notice the formation of silicic acid. How do you reconcile the first reaction in this experiment with the last?

104. Hydro-fluo-silicic acid, silicic acid, and their salts (elective; hood).—Arrange an apparatus as shown in

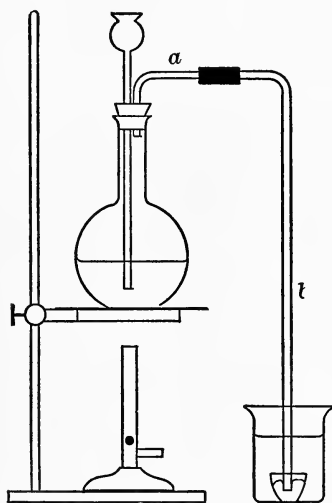


FIG. 22.

the cut. In a 300 c.c. flask put a mixture of 15 grammes of finely ground sand and 10 grammes of finely ground calcium fluoride, CaF_2 . Pour in enough concentrated sulphuric acid to make a thin paste. Moisten the powder all through with the acid by shaking the flask. Connect the flask with a dry tube (a). The end of the dry tube (b) dips beneath the surface of a little dry mercury contained in a No. 00 porcelain crucible which stands in a beaker of 150 c.c. capacity. Nearly fill the beaker with water without letting any of it enter the tube. Heat the flask carefully, and note the formation in the beaker of the jelly-like silicic

acid. Stir occasionally, and continue to pass the gas until the water is well filled with the jelly. Disconnect the apparatus. Remove the crucible and mercury, and pour the mercury into the bottle for impure mercury. Do not pour it into the sink or slop jar.

Filter the contents of the beaker, keeping both the jelly (*c*) and the filtrate (*d*). Wash the jelly twice on the filter. Heat a portion of it with a strong solution of sodium hydroxide, using as little as is necessary to dissolve it. To the solution add hydrochloric acid drop by drop and note the precipitation of the silicic acid.

Dry the rest of the precipitate by pressing between filter papers, and heat slightly in an evaporating dish until it is all changed to a white powder of silica. To make it quite pure, it must be washed with water.

Test the filtrate (*d*) with blue litmus paper. To a portion of it add a solution of barium chloride. To another portion add a solution of potassium nitrate or chloride. Label the last formed precipitate and let it stand. Notice the precipitate and its character, and then shake the test tube containing it.

Explain the whole process and write the equations.

BASIC ELEMENTS

The experiments from this point on require chemically pure (C. P.) reagents and distilled water. Before this crude acids and alkalis may be used, except where pure

reagents are especially called for. If crude acids have been used, the bottles should now be emptied, cleaned, and filled with pure acids.

MAGNESIUM

105. Burn a piece of magnesium ribbon about 15 cm. long. What is formed? Dissolve it in dilute hydrochloric acid. Evaporate to dryness, and dissolve the residue in water. To portions of this solution add solutions of sodium carbonate, of sodium phosphate, Na_2HPO_4 , of sodium sulphate, of potassium nitrate, and of potassium hydroxide. Why in some cases are precipitates formed and not in others?

106. Put a piece of magnesium ribbon in a small beaker of water. Fill a test tube with water and invert it in the beaker. Add to the water in the beaker a little hydrochloric acid, and collect the gas in the test tube. What is the gas? What becomes of the metal? Evaporate the solution to dryness, and repeat the experiments in (105). In what three ways can magnesium chloride be made?

ZINC

107. Heat a few pieces of zinc with a strong solution of sodium hydroxide. What gas escapes? What is there in the solution? From your previous experience, what acids will dissolve zinc?

108. Heat a little zinc oxide. Notice the color change on heating.

109. Make a solution of zinc sulphate. Add to portions of it hydrogen sulphide, sodium phosphate, and sodium hydroxide in small quantity and then in excess. (Compare 107.)

Are the precipitates bases or salts?

SILVER*

110. Test a solution of silver nitrate with the following reagents: sodium chloride, sodium phosphate, potassium chromate, K_2CrO_4 , and potassium iodide. In each case test the solubility of the precipitate in ammonia water. Set the precipitate of the chloride where it will be in the light, and note the change in color.

How much silver nitrate will be required to produce 235 grammes of silver iodide?

111. **Silver oxide.** — To 2 c.c. of ordinary solution of silver nitrate add an excess of caustic soda solution. Filter off the precipitate and wash it, and dry slightly by heating it in an evaporating dish. Then heat strongly. What is formed? Dissolve the residue in nitric acid. Can you tell by the reaction whether a metal or an oxide is present? Write out all equations.

* Put all silver wastes into the bottle placed for that purpose.

112. Into 5 c.c. of nitrate of silver solution, in a test tube, put about a gramme of mercury (a drop about the size of a small pea), and set the tube where it will be undisturbed. Notice the metallic crystals of an amalgam of silver and mercury.

COPPER

113. To a solution of copper sulphate add ammonia water, drop by drop, and finally in excess. Filter after adding excess. To another portion add sodium hydroxide in moderate quantity, then in excess, and boil. (Compare 111.) To another portion add hydrogen sulphide. To another portion add potassium ferrocyanide, $K_4Fe(CN)_6$. Dilute a few drops with half a litre of water and add a few drops of potassium ferrocyanide. Write the equations and explain.

114. Into a solution of copper sulphate put a piece of clean iron. Explain what you see.

115. Cuprous chloride (elective). — Into a 200 c.c. flask put 10 grammes of metallic copper (copper turnings) and 50 c.c. of concentrated hydrochloric acid. Then add 1 c.c. of concentrated nitric acid, and, after the first rapid action is over, boil gently for fifteen minutes. Pour out into half a litre or more of water, and, after the white cuprous chloride has settled, pour off the liquid and wash by decantation until the wash water is no longer blue.

Dissolve a part of the precipitate in ammonia water. Dissolve a part in concentrated hydrochloric acid and pour this solution again into a large amount of water. To a part of the precipitate add an excess of sodium hydroxide and boil. Compare with the action of sodium hydroxide on a cupric salt.

Why in making cuprous chloride should not all of the copper be dissolved?

MERCURY*

What is the weight of 1 c.c. of mercury?

116. Mercurous compounds. — Warm about a gramme of mercury with dilute nitric acid, not adding enough acid to dissolve it all.

To a portion of this solution add the following reagents: hydrochloric acid, potassium hydroxide, potassium iodide, and hydrogen sulphide.

117. Mercuric compounds. — To a larger portion of the solution of mercurous nitrate made in (116), add more nitric acid and boil until it fails to give a precipitate with hydrochloric acid. Test the solution thus made with the reagents used above. Compare the phenomena caused by the same reagent in the two cases.

From 1 gramme of mercury, how much mercurous

* Put all mercury or solutions containing it into the bottle provided for that purpose. Do not pour it into the sinks or jars.

nitrate can be made, and, from this, how much mercuric iodide can be made?

118. Into a solution of mercuric chloride put a piece of clean copper. Notice the deposit on the copper. Rub it lightly with the finger. Write the equation. Compare with (112) and (114), and arrange the metals, iron, silver, copper, and mercury, in the order in which they displace one another in their salts.

TIN

119. Stannous compounds. — Dissolve about a gramme of tin in concentrated hydrochloric acid. (By adding a few drops of a solution of platinum chloride or of copper chloride, the reaction can be greatly hastened.) Dilute part of the solution and make the following tests. To part of the solution add a little hydrochloric acid, and then drop by drop a solution of potassium permanganate, KMnO_4 . Why is it decolorized? Add some of the tin solution drop by drop to a solution of mercuric chloride. Continue to add until no further change in color can be seen. Explain. To a portion of the solution add hydrogen sulphide. Notice the color of the precipitate.

120. Stannic compounds. — Add to a portion of the stannous chloride solution a little hydrochloric acid, then nitric acid, and boil. Dilute a sample and add to it potassium permanganate. If it decolorizes this, add more nitric

acid and boil again. When it will no longer decolorize the permanganate, test it with mercuric chloride and with hydrogen sulphide as above. What chemical change has the nitric acid produced? Note the difference in the actions of the two tin solutions toward reagents.

121. Treat a little tin in a test tube with a mixture of equal parts of water and concentrated nitric acid. What tin compound is made?

122. Anhydrous stannic chloride (elective; hood). — In a *dry* 100 c.c. tubulated retort put 10 grammes of granulated tin. Set up an apparatus for making chlorine as in (8), but using twice the quantity of material. Dry the chlorine by passing it through sulphuric acid and let it enter the retort through a cork fitted to the tubulus of the retort. (See Figs. 18 and 19.) Heat the retort until the tin melts and collect the stannic chloride, which distils over, in a *dry* side-neck distilling flask used as a receiver. When the tin is all gone, disconnect the apparatus and distil the product in the side-neck flask, observing on a thermometer the temperature of its boiling and the steadiness with which it maintains that temperature. Collect in a *dry* bottle and cork with a rubber stopper to keep it from the moisture of the atmosphere. Cautiously pour a little into an equal quantity of water and let it cool. Dissolve in more water, and make with the solution the tests for stannic compounds.

LEAD

123. Dissolve some lead in nitric acid. (a) Dilute the solution and test with sulphuric acid, hydrogen sulphide, and potassium chromate.

(b) To another portion add hydrochloric acid. Boil with water the precipitate formed, and cool the solution obtained under the hydrant in a test tube or flask.

(c) To another portion add potassium hydroxide, drop by drop, and then in excess. To this solution, whose content you should know before proceeding further, add solution of sodium hypochlorite, NaClO , or pass through it chlorine gas. What is the precipitate?

Heat some litharge, PbO , with hydrochloric acid. Heat some lead peroxide, PbO_2 , with hydrochloric acid. Cautiously notice the odor of the vapors given off.

Treat each of the three oxides of lead, litharge, PbO , lead peroxide, PbO_2 , and red lead, Pb_3O_4 , with nitric acid.

In a dilute solution of lead nitrate in distilled water suspend by a string a piece of zinc, and set the vessel where it will not be disturbed.

NICKEL

124. Determine the conduct of nickel sulphate toward the reagents: (1) ammonia water, (2) ammonia water and hydrogen sulphide, (3) caustic soda, (4) caustic

soda and potassium cyanide, (5) caustic soda and sodium hypochlorite.

COBALT

125. With a solution of the chloride or nitrate use the reagents mentioned in the preceding experiment. Make a table to show the resemblances and differences.

126. Make a borax bead. Dissolve in it while hot a little of a cobalt compound, and observe the color of the bead when cool. (See 100.)

127. Smear a little of a solution of a cobalt salt on a piece of paper, and cautiously warm it above the burner. Note the change of color and explain. Treat a crystal of cobalt chloride with strong hydrochloric acid.

IRON

128. Ferrous compounds. — (a) Put about 10 grammes of metallic iron in a flask, add 100 c.c. of water, and pour in enough concentrated sulphuric acid to start a brisk evolution of hydrogen. While hydrogen is still being evolved, pour out portion after portion, and test each with one of the following reagents: (1) potassium ferricyanide, $K_3Fe(CN)_6$, (2) potassium ferrocyanide, $K_4Fe(CN)_6$, (3) potassium sulphocyanate, $KCNS$, (4) freshly prepared hydrogen sulphide solution followed by ammonia water.

(b) To another portion add ammonia water, note the color of the precipitate. Then shake with air, and note the color of the precipitate. Let this test tube stand, and note next day the color of the precipitate, especially where it has come into most ready contact with the air. Why the changes of color?

(c) Filter the remainder of the sulphate solution. What may the black residue be? Evaporate to a small bulk, but not to dryness, and set away to cool. What is the common name of the salt crystallizing out?

129. Ferric compounds. — Make a dilute solution of a ferric salt, ferric chloride, FeCl_3 , or ferric potassium alum, $\text{Fe}_2(\text{SO}_4)_3 + \text{K}_2\text{SO}_4 + 24 \text{H}_2\text{O}$, and test it with the reagents used with the ferrous salts. What is the precipitate caused by hydrogen sulphide alone. Make a table to contrast the two sets of reactions.

130. Anhydrous ferric chloride (elective; hood).—Into a piece of hard glass tubing, with an internal diameter of 1 cm. and a length of about 30 cm., put a loose tangle of clean iron wire about 10 cm. long. Connect the tube with an apparatus for generating *dry* chlorine. Support the tube in a horizontal position, and while the chlorine is passing heat the iron gently. Fumes of ferric chloride will soon appear and condense in the cooler portions of the tube in crystalline lustrous scales. While the chlorine is passing, part of the chlorine may be sub-



lined by heat into a dry test tube placed over the end of the hard glass tube, and may be preserved by drawing out and sealing off the upper end of the tube with the blast lamp.

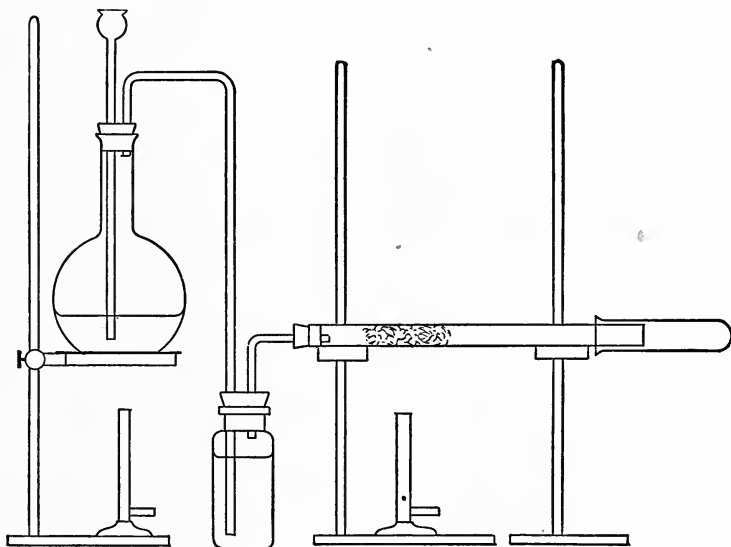


FIG. 23.

Dissolve part of the chloride in water, and apply some of the most striking tests used above for ferric salts.

ALUMINUM

131. Treat a little aluminum with concentrated hydrochloric acid. Treat a little with concentrated nitric acid. Treat some with solution of caustic soda. What gas is given off? What is contained in the solution?

132. To a solution of aluminum chloride add, a little at a time and finally in excess, a solution of potassium hydroxide. Write equations showing the reactions. (See 131.)

133. To a solution of aluminum chloride add sodium carbonate. Notice effervescence of carbon dioxide and formation of a precipitate. What is the precipitate, and why is not a carbonate formed?

CHROMIUM AS A BASIC ELEMENT

134. Dissolve some chromium hydroxide, $\text{Cr}(\text{OH})_3$, in hydrochloric acid. What is formed? To what general class of substances does chromium hydroxide belong? To what class of substances does the compound formed belong? How else could you make it?

135. To the solution made above add sodium hydroxide a little at a time. What is formed? Add an excess. (Compare 132.) Then boil.

136. To a solution of chrome alum $\text{K}_2\text{Cr}_2(\text{SO}_4)_4$, add a solution of sodium carbonate. Notice the effervescence. (Compare 133.)

Notice the similarity, to this point, of chromium and aluminum.

CHROMIUM AS AN ACID-FORMING ELEMENT

137. To a solution of 5 grammes of potassium chromate, K_2CrO_4 , in 10 c.c. of water, add 10 c.c. of concentrated sulphuric acid. What separates on cooling? What corresponding compound of sulphur is there? If this compound or the corresponding compound of sulphur unites with water, what kind of a substance will be formed? To what class of substances do the crystals and the corresponding sulphur compound belong?

138. To small portions of a solution of potassium chromate add in turn the reagents: (1) silver nitrate, (2) lead acetate, (3) barium chloride. To what class of substances do the potassium chromate and the precipitates formed belong, and of what substance are they derivatives?

139. Chromium salt to chromate. — To a solution of chrome alum add a solution of sodium hydroxide until no more precipitate is formed. Bring to boiling, and add solution of sodium hypochlorite, $NaClO$, until a clear yellow solution is obtained. To small portions of this solution add in excess: (1) silver nitrate, (2) lead nitrate, (3) barium chloride. (Compare 138.) Explain the whole process and write equations.

140. Chromate to salt of chromium. — Grind to a fine powder 10 grammes of potassium chromate, K_2CrO_4 . Put into a half-litre flask and add a mixture of 40 c.c. of

water and 10 c.c. of concentrated sulphuric acid. Add to the mixture 10 c.c. of alcohol *a little at a time*. (Caution!) What color has the solution? If the color is too dense to be apparent, dilute a portion. Set the solution aside to cool. The next day examine it. If crystals have not yet formed, set away for two days more. Dissolve some of the crystals in water. What is the color of the solution? Compare with the color of the original solution and explain the difference.

MANGANESE

141. To a solution of manganese chloride add caustic soda solution. Note the color of the precipitate and divide the contents of the tube into two parts. To one portion add a solution of sodium hypochlorite. Let the other portion stand exposed to the air. What change of color is seen in each case, and to what is it due?

142. Test a solution of manganese chloride with: (1) ammonium sulphide, (2) sodium carbonate, and (3) ammonia water.

143. Make a loop of platinum wire and in this a bead of potassium carbonate, K_2CO_3 . Touch this bead to any compound of manganese, taking care not to take up too much of the compound. Heat again, with the oxidizing or outer portion of the Bunsen flame, and then see whether the bead on cooling has a green

color. If it has not, add to the bead a fragment of potassium nitrate, or better touch the bead to the stopper of the nitric acid bottle, moist with the acid, and heat again. Make the experiment again to see how little of the manganese compound is necessary. To what is the green color due? (See 144.)

144. Potassium manganate and permanganate.—Heat together in an iron dish 10 grammes of solid potassium hydroxide and 5 grammes of manganese dioxide. When fused, add 5 grammes of potassium nitrate. Continue heating until a sample of the mass, taken out with a glass rod, has a fine dark-green color. When the mass is cool, dissolve in 100 c.c. of water, and filter through a plug of glass wool. Boil the solution in a beaker, and add dilute sulphuric acid, a drop at a time, until the color becomes a deep red. Filter through glass wool. What is the precipitate? What is contained in the solution? Write the equations.

To the solution add dilute sulphuric acid and then a solution of ferrous sulphate. To another portion add sulphuric acid and a solution of oxalic acid. What becomes of the reagents added? What kind of action has the permanganate upon them?

PART II

VERIFICATION OF QUANTITATIVE LAWS

All data should be preserved in note books and not on loose pieces of paper. Where an experiment is repeated, report the results not satisfactory with the probable cause of the difficulty, as well as the satisfactory results.

In all cases calculate the results so as to have them directly comparable with the results obtained by others. For example, in the weight problems let the results appear as percentages of the original weights.

WEIGHT RELATIONS

1. Constant proportions. —(a) Dissolve about 20 grammes of pure sodium hydroxide in about 200 c.c. of distilled water. Carefully measure into a No. 0 evaporating dish, with a 10 c.c. pipette, 30 c.c. of this solution. Exactly neutralize it with dilute, *pure* hydrochloric acid. Evaporate the neutral solution to dryness on the water bath. Heat with the Bunsen burner carefully at first, and finally with the full power of the burner until it loses

no more in weight. Weigh and note the gain. (Perform 1, *b* at the same time.)

(*b*) Carefully measure as before 20 c.c. of sodium hydroxide solution, and add an excess of hydrochloric acid: that is, more acid than enough to neutralize it. Evaporate, and heat, and weigh as before.

Compare weight gained in this case with that in 1 (*a*).

How do these experiments illustrate the law of constant proportions?

2. Constant proportions. — (*a*) Weigh a No. 0 porcelain crucible with its cover. Brighten about a metre of magnesium ribbon with sandpaper. Wipe off the loose particles from the brightened ribbon. Make it into a coil, press it firmly down into the crucible and weigh again with the cover. Heat carefully on a pipe-stem triangle with a clear-burning Bunsen burner. Hold the burner with one hand, and the cover of the crucible with the other, by means of a stiff wire with a loop in the end, which is passed through the ring on the crucible cover.

When the crucible begins to be red hot, the magnesium will start to oxidize. If the metal begins to burn, or fumes appear above the mouth of the crucible, put on the cover, take away the burner, and wait until the glow of the combustion can no longer be seen through the porcelain. Keep on heating until there is no longer a tendency to burn or glow without external heat, and

finally heat strongly with the cover off and the crucible inclined so as to cause a draft of air through it. (The cover should be carefully handled and kept, to be weighed with the crucible, as there may be some magnesium oxide adhering to it.)

Cool and weigh with the cover. Heat again with the cover off and the crucible inclined. Again cool and weigh. Repeat if there is a gain in weight.

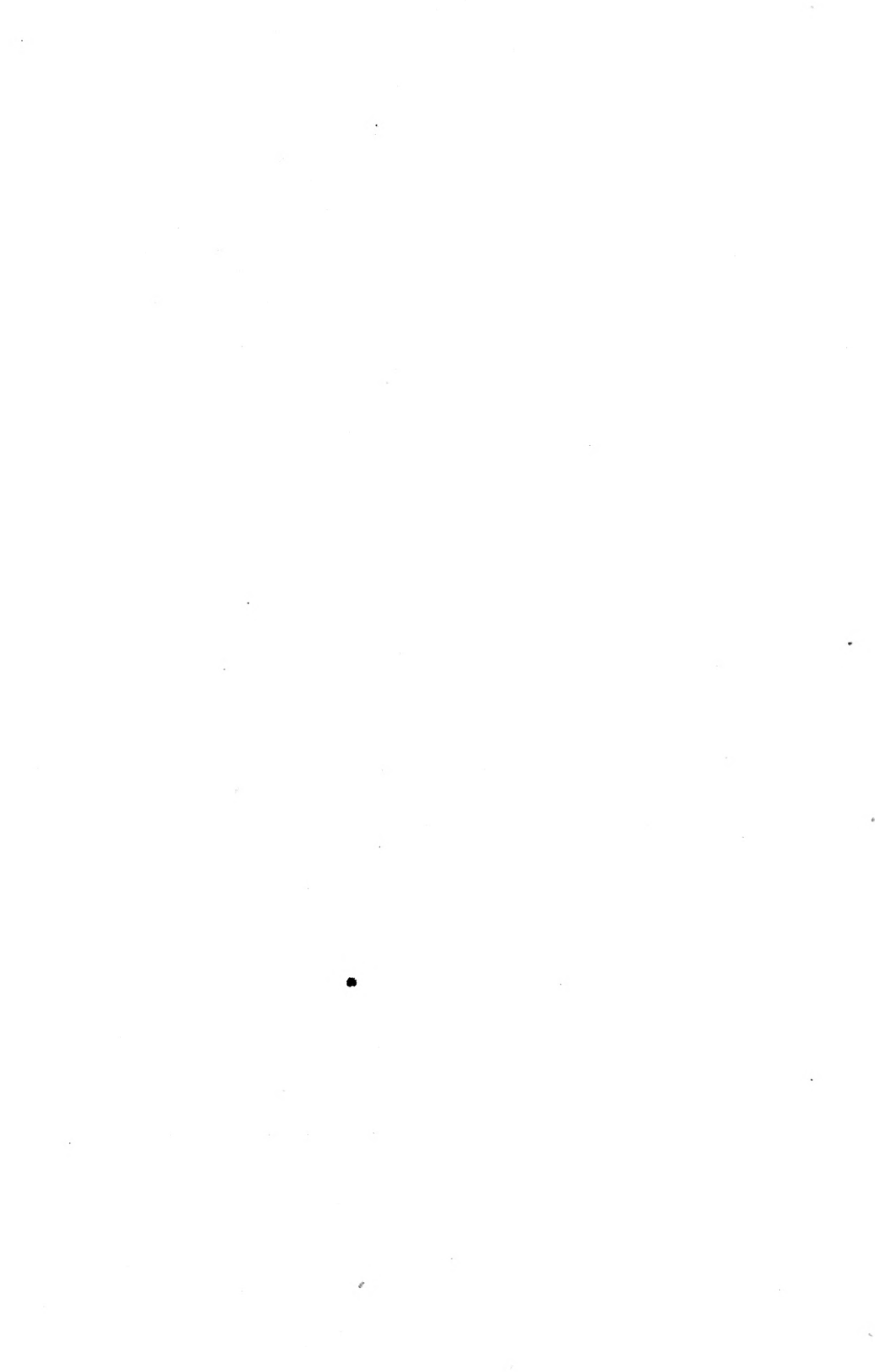
What percentage has the magnesium gained?

(b) Weigh a No. II crucible. Prepare as before about half a metre of magnesium ribbon. Press into the crucible and weigh. Cover the metal with distilled water.

Add, a few drops at a time, concentrated *pure* nitric acid, and after each addition cover the crucible with a clean watch glass. When the metal is dissolved and no more red fumes are evolved, rinse the watch glass into the crucible with a wash bottle. Evaporate to dryness on a water bath, then heat with the burner carefully at first and then strongly until no more red fumes are evolved.

Cool and weigh. Repeat heating and weighing until there is no further loss in weight.

Write the equations indicating the reactions. What percentage of gain has the magnesium made? Compare the gain in this experiment with that in 2 (a). Compare the deductions from this experiment with those from 1 (a) and (b).



Multiple Proportions

3. (a) Make a tube of hard glass about 1 cm. in internal diameter and about 15 cm. in length, closed at one end so that there are no thick bunches of glass and well annealed.

Weigh the tube and put into it 2 or 3 grammes of pure potassium chlorate, and weigh again. Close the mouth of the tube not too tightly with a plug of long-fibre asbestos, heating the asbestos in the flame just before putting it into the tube.

Weigh again. Now heat the chlorate carefully at first and then strongly until no more gas is evolved. Finally bring each part of the tube to a high heat to decompose any of the salt that may have spattered or have been carried to the asbestos as spray. Cool and weigh.

What percentage of the original chlorate is the loss in weight?

What percentage of the residual chloride is the loss in weight?

Remove the residue from the tube and confirm the supposition that it is potassium chloride by the following tests. Observe the color imparted to the Bunsen flame when a little of the substance is put into the flame on a clean platinum wire. Try the action on it of concentrated sulphuric acid. Try the action of silver nitrate solution on its solution.

(b) Repeat 3 (a) using potassium perchlorate, KClO_4 ,



instead of potassium chlorate. Make calculations and test residue as before.

With a fixed amount of potassium chloride how much oxygen was combined in each case and what is the percentage ratio of the amounts of oxygen? Tell how the experiment verifies the law.

4. Volume composition of hydrogen chloride. — Thoroughly dry a mortar. Pass the handle of the pestle tightly through a piece of cardboard which will protect the hand in the next operation. Put into the mortar about 10 c.c. of dry mercury, and under the draft cupboard add a clean piece of sodium of a bulk of about 1 c.c. With the pestle press the sodium against the bottom of the mortar, when it will unite with incandescence with the mercury to form an amalgam. Stir with the pestle and, if the amalgam is not to be used immediately, preserve it in a tight, *dry* bottle. Make hydrogen chloride by letting strong sulphuric acid fall, drop by drop, from a separatory funnel into concentrated hydrochloric acid. Dry the gas by letting it pass through strong sulphuric acid. Why does the solution liberate the gas in this way? How else can hydrochloric acid gas be prepared?

Have a tube closed at one end of 15 mm. diameter and 60 cm. length. Thoroughly clean and dry it by warming it over the flame of the Bunsen burner, at the same time causing air to pass through it by exhausting

with a filter pump through a long tube passed to the closed end of the larger tube.

Fill the tube with hydrochloric acid gas by connecting a long tube, which passes to the bottom of the larger tube, with the generating flask and collecting by displacement of air. Let the gas pass for some time to thoroughly displace the air.

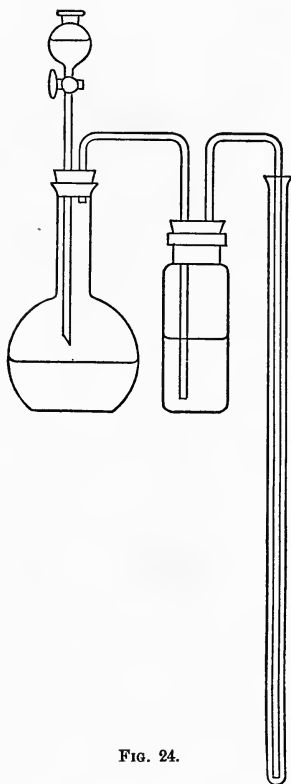


FIG. 24.

When the tube is filled, take out the narrow tube slowly. Pour into it the sodium amalgam. Close quickly and tightly with the thumb slightly moist, and shake to decompose the acid.

Invert the tube in a basin of water and withdraw the thumb. Now transfer to a tall cylinder of water, bring the water on the inside and outside to the same level and mark, with a tight rubber band, the point on the tube separating the gas from the water.

Measure with a c.c. graduate the space occupied by the gas and the volume of the whole tube. Remember that the volume of the hydrochloric acid gas was dry, and that the hydrogen remaining is measured over water, and correct the volume of the hydrogen for aqueous

tension. (See appendix.) Compare the volume of the original gas with that of the residual gas. Repeat until concordant results are obtained.

Express by an equation the change that has taken place, and explain the change in volume.

5. Volumetric composition of ammonia. — *In ammonia one volume of nitrogen is combined with three volumes of hydrogen.*



FIG. 25.

Fit to the long tube used in the last experiment a rubber stopper, through which a short-stemmed separatory funnel is passed, in such a way that the end of the funnel is even with the end of the cork.

Fill the dry tube with chlorine as it was filled in the last experiment with hydrochloric acid gas. Having the stop cock of the funnel closed, fill the tube of the funnel with water and fix the cork in the tube. Pour into the funnel an excess of strong ammonia water. Open the stop cock a little, and let flow in gradually a little more ammonia water than will react. Follow this by dilute sulphuric acid, and finally let as much water flow in as will. Take care that at no time is air drawn through the funnel. Mark with a rubber band the height of the water.

Measure the space occupied by the gas and compare with the volume of the tube.



What reactions have taken place? How many volumes of hydrogen are required to unite with one volume of chlorine to make hydrochloric acid? (See 4.) Then how much hydrogen would be required to unite with the chlorine which filled the tube? What has become of the chlorine? Where did the residual gas come from? What was combined with it, and what volume in the free state would it occupy?

Give the general conclusion.

5 (a). *Two volumes of ammonia yield one volume of nitrogen.*—Fill the long tube of the last experiment, after drying it thoroughly, with dry ammonia gas. The gas must be dried by passing it over coarsely powdered quicklime, and may be made by *warming* concentrated ammonia water.

Place the stopper and funnel as in the last experiment, and let flow in a little at a time a solution of sodium hypochlorite (chlorinated soda), taking care to shake well after each addition of the reagent, and to give time for the reaction to complete itself before adding more. When enough hypochlorite has been added to decompose the ammonia, let water flow in as much as will, and compare the volume of the residual gas with the total volume of the tube. Here, also, it should be remembered that the volume of ammonia, which filled the tube at first, was dry while the residual gas is measured wet. Apply a correction.

Write equation, interpret results, and explain by the molecular theory the volume relations.

6. Composition of water. — Prepare an apparatus for the generation of hydrogen. Provide a hard glass tube about 15 cm. in length and of an internal diameter of from 7 to 10 mm. Put into it about 2 grammes of cupric oxide, CuO . The oxide should be heated with the full power of the burner, in a porcelain crucible, just before putting it into the tube.

Near each end of the tube place a loose plug of freshly ignited asbestos fibre. The rubber stoppers in the ends of the tube should have the diameter at the smaller end the same as the inner diameter of the tube. Push them in tightly to prevent water collecting between the stoppers and the sides of the tube. Weigh the tube with the contained copper oxide, asbestos, and stoppers.

Pass hydrogen from the generator through a tube (*b*) containing granulated calcium chloride, then over the copper in the tube (*c*), and through a second calcium chloride tube (*d*) which has been weighed. Do not let the reaction in the generator become so rapid that much heat is perceptible, since then there is danger of more water coming over than can be taken up by the first calcium chloride tube.

When the whole apparatus is filled with hydrogen (test the evolved gas), heat the copper oxide and continue heating until nearly all of the black oxide has been

changed to red copper. Now heat gently and continue to pass the hydrogen for some time after all water has apparently disappeared from the copper oxide tube.

Cool while passing the hydrogen. Disconnect (a) and displace the hydrogen by blowing through (b). Weigh (c) and (d).

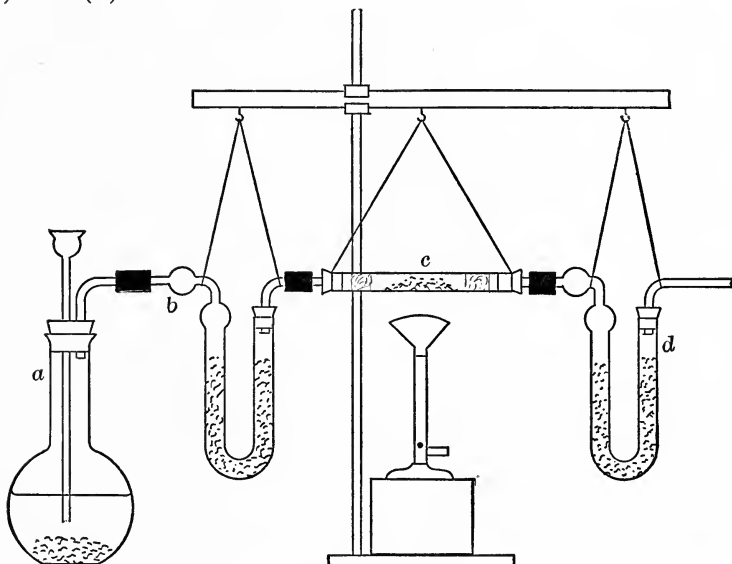


FIG. 26.

The loss of weight of (c) represents the oxygen necessary to form the amount of water represented by the gain in weight of (d).

Calculate the percentage of oxygen and hydrogen in water.

7. Hydrogen equivalent of zinc. — Place in a beaker a No. 00 crucible. Weigh a piece of *pure* zinc (about 0.1

gramme) to the nearest milligramme. Place the zinc in the crucible and fill the beaker with water until it stands somewhat higher than the top of the crucible. Fill a

50 c.c. stop-cock eudiometer* with water and invert it in the crucible. Pour into the funnel of the eudiometer some pure hydrochloric acid to which two or three drops of solution of platinum chloride have been added. Let the acid flow in, a little at a time, after placing the eudiometer so that the gas will pass into it.

When no more gas is evolved, transfer the tube to a cylinder of water, bring the water inside and outside to the same level, and read the volume. (Do not hold the tube with the warm hand.)

Take the barometric pressure and the temperature of the water in the cylinder, and, after reducing to standard conditions of pressure and temperature, calculate the weight of the hydrogen. (See appendix.) Calculate the ratio $H : Zn :: 1 : x$.

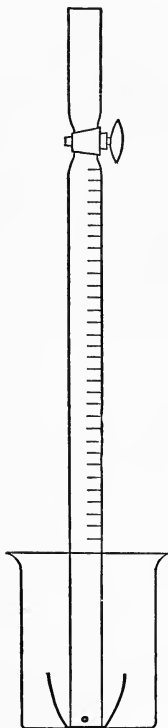


FIG. 27.

8. Oxygen equivalent of zinc.—In a large weighed porcelain crucible (R. B. Porcelain No. III) dissolve about

* This elegant piece of apparatus was brought into regular use and was probably invented by Professor W. W. Daniells. An apparatus which will answer in this experiment may be made from an open tube and a separatory funnel.

30 grammes of *pure* zinc by covering it with water, and adding drop by drop pure concentrated nitric acid. Keep the crucible covered with a watch glass during the reaction. Rinse the watch glass into the crucible with a wash bottle, and evaporate the solution to dryness on a water bath. Heat carefully with the Bunsen flame until the whole mass shows a yellow color, and then ignite repeatedly with the blast lamp until there is no more loss of weight.

From the increase in weight of the crucible find the amount of zinc oxide, and from this calculate the ratios $\text{Zn} : \text{ZnO}$ and $\text{Zn} : \text{O}$.

Compare with the result of the last experiment to get the hydrogen equivalent of oxygen, $\text{H} : \text{O}$. (Compare also with 6.)

9. Vapor density and molecular weight. — If the percentage of each element in a compound is divided by the atomic weight of the element, the resulting quotients will have the same ratio as that between the number of atoms in the molecule. A compound having the number of atoms in this ratio has the percentage composition of the substance analyzed, but the substance may have a molecular weight twice or three times as great as that indicated by the simple ratio existing between the quotients above. There is a substance whose true molecular formula is C_2H_2 , and another substance whose true formula is C_6H_6 , in both of which the percentage composition is the same.

To distinguish between substances having the same composition we may with volatile substances determine their vapor densities, and from this, by the law of Avogadro, find their molecular weights and true formula.

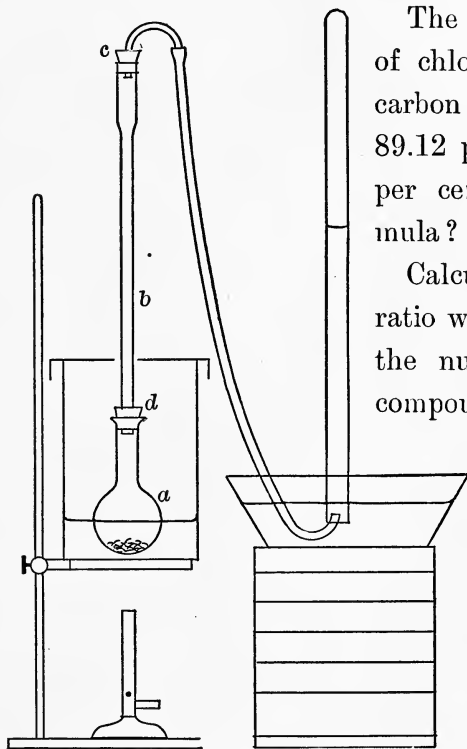


FIG. 28.

The percentage composition of chloroform is by analysis, carbon 10.04 per cent, chlorine 89.12 per cent, hydrogen 0.84 per cent. What is its formula?

Calculate first the simple ratio which can exist between the number of atoms in a compound having the given composition. What molecular weights will satisfy this ratio?

Determine the vapor density according to the method of Victor Meyer as follows: The apparatus consists of a round bottom flask

(a) of 300 c.c. capacity, in the bottom of which is placed a layer of asbestos or glass wool. Provide the flask with a well-fitted stopper to which is fitted a tube (b) of about 1 cm. internal diameter and 50 cm. length. (The inner tube of a Liebig's condenser.) To the upper



end of the tube a stopper is fitted, to which is attached a rubber tube to lead to the eudiometer the air which is expelled. Push in firmly the stopper (*d*), and also push in firmly the stopper (*c*) and mark the position of its lower end with a file. Support the apparatus in a vessel containing water (a tin calcium chloride can or the lower part of an ether can), and heat the water to boiling. When no more air is expelled, place the end of the rubber tube under the eudiometer, remove (*c*) and drop in a little glass tube containing about 0.3 gramme of chloroform, weighed while the apparatus is heating. After dropping in the chloroform, *immediately* replace the stopper and collect in the eudiometer by displacement of water the air expelled by the chloroform. When no more air is expelled lift (*c*).

Bring the eudiometer into a cylinder of water and read the volume, the temperature, and the barometric pressure.

The chloroform vapor is heated to the same temperature that the air had before it was expelled. In escaping, the air is cooled to the room temperature. Hence, the volume of air measured is the volume which would be occupied by the chloroform, if it could be volatilized under that pressure and at that temperature.

Reduce the volume read to standard conditions, noting that the air is measured over water. Calculate the density of chloroform as compared to hydrogen, and from this the true molecular weight.

10. Molecular weight by chemical means. — Certain substances, the molecular weights of which are desired, cannot be volatilized without decomposition, and some other method besides that by means of the vapor density must be used. A purely chemical method, which may be employed in the case of organic acids, will be given here.

By analysis, the ratio between the number of atoms is determined as in the preceding experiment. The acid is known by experiment to be a monobasic-acid, that is, it forms but one series of salts, or according to theory there is but one hydrogen atom replaceable by metals.

When the silver salt is heated, all is driven off except a residue of metallic silver. The equivalent of silver is 108, that is 108 parts of silver are capable of replacing one part of hydrogen. In the silver salt one part of hydrogen has been replaced by 108 parts of silver, and the molecular weight of the salt is the same as that of the acid plus (108 - 1).

As an illustration, take the case of lactic acid. By analysis, lactic acid contains 40.16 per cent of carbon, 6.69 per cent of hydrogen, and 53.14 per cent of oxygen, and its atomic ratio is $C:H_2:O$. The true formula may be CH_2O , $C_2H_4O_2$, $C_3H_6O_3$, or some higher multiple of CH_2O . Lactic acid is a monobasic acid, and only one of its hydrogen atoms can be replaced by silver.

By igniting its silver salt there is a residue of metallic silver. The part driven off represents all of the molecule

of the salt, except one atom of silver, or all of the molecule of the acid, except the one atom of hydrogen. If we refer all these weights to the atomic weight of hydrogen as a standard, we can find the sum of the atomic weights of the part of the salt not silver, and by adding one unit to this sum we will obtain the molecular weight of the acid.

Let (a) be the weight of the silver salt.

Let (b) be the weight of the residue of silver.

Let (M) be the molecular weight of the acid.

$$a - b = \text{wt. driven off.}$$

$$\frac{b}{108} = \frac{a - b}{M - 1}, \quad \therefore Mb - b = 108a - 108b.$$

$$M = \frac{108a - 107b}{b} = 108 \frac{a}{b} - 107.$$

In the case of lactic acid 100 parts of silver salt yield 54.9 parts of silver, and from this, by use of the mathematical formula, we find

$$M = 108 \frac{100}{54.9} - 107 = 90.$$

That is, the true molecular weight is 90, or lactic acid has the formula $\text{C}_3\text{H}_6\text{O}_3$, or three times that indicated by the atomic ratio.

PROBLEM. — Acetic acid has the same percentage composition as lactic acid, or in percentages, C 40.16, H 6.69,

and O 53.14. What is the atomic ratio? And what is the molecular weight?

Make silver acetate in the following manner: To a solution of 2 grammes of silver nitrate in 40 c.c. water add a strong solution of ammonium acetate as long as a crystalline precipitate forms. Filter and wash the precipitated silver acetate four or five times by filling the filter with water and letting it run off. (Save filtrate and wash water with silver residues.) Let the salt drain. Press it between filter papers and let it remain in a dry place protected from the light for twelve hours.

Weigh a small crucible and in it weigh about half a gramme of the salt. Heat the crucible gently and from the side. Gradually heat more intensely and finally with the full power of the burner. Cool, weigh, and calculate the amount of silver residue.

From the data obtained calculate the molecular weight of acetic acid and its true formula.

APPENDIX

CALCULATION OF GAS VOLUMES

When a gas whose volume is V at 0°C. has its temperature raised, the pressure (P) being kept constant, its volume increases $\frac{1}{273}$ of its volume at 0°C. for each degree increase of temperature. That is, volume at $0^\circ = V_0$.

$$\text{Volume at } 1^\circ, (V_1) = V_0 \left(1 + \frac{1}{273}\right),$$

$$\text{Volume at } 2^\circ, (V_2) = V_0 \left(1 + \frac{2}{273}\right),$$

$$\text{Volume at } 3^\circ, (V_3) = V_0 \left(1 + \frac{3}{273}\right),$$

$$\text{Volume at } t^\circ, (V_t) = V_0 \left(1 + \frac{t}{273}\right).$$

The fraction $\frac{1}{273}$ is expressed decimally .03667 and is commonly indicated by the Greek letter α . Then if V_0 equals the volume at 0° , $V_t = V_0(1 + \alpha t)$, $V_0 = \frac{V_t}{1 + \alpha t}$.

The product of the volume of a gas and the pressure to which it is subjected is a constant. Then if V_s = volume at 0°C. and the standard pressure, 760 mm. of mercury, and V_0 = volume of the same gas at 0°C. and pressure P ,

$$760 V_s = V_0 P, \quad V_s = \frac{V_0 P}{760}.$$

To reduce for both pressure and temperature combine these equations.

$$V_s = \frac{V_t P}{760 (1 + \alpha t)}$$

If the gas is collected over water, or if it is in contact with water, it will be saturated with water vapor. This water vapor exerts a pressure or tension which varies with the temperature. This tension for various temperatures has been experimentally determined and may be found in the accompanying table.

If the surface of the water inside and outside the vessel is at the same level, the barometric pressure (B) outside is balanced by the actual pressure of the gas (P) added to the aqueous tension (p) at the temperature of the room, *i.e.* $B = P + p$, and the pressure which the gas itself has when it occupies the volume V will be $P = B - p$.

Introducing this value in the last equation we have the formula for reducing to standard conditions of pressure and temperature and correcting for aqueous tension,

$$V_s = \frac{V_t(B - p)}{760(1 + \alpha t)}, \text{ where}$$

V_s = Volume at standard conditions.

V_t = Volume as observed.

B = Barometric pressure.

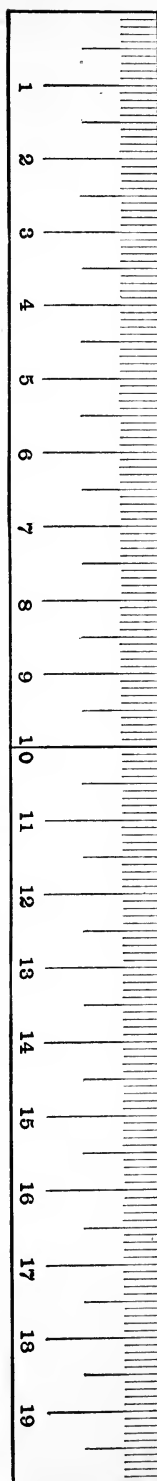
p = Aqueous tension at observed temperature.

α = Cubic expansion of gases.

TENSION OF AQUEOUS VAPOR FROM
0° TO 30° C.

TEMPERATURE	TENSION IN MM.
0° C.	4.60
1° C.	4.94
2° C.	5.30
3° C.	5.69
4° C.	6.10
5° C.	6.53
6° C.	7.00
7° C.	7.49
8° C.	8.02
9° C.	8.57
10° C.	9.17
11° C.	9.80
12° C.	10.46
13° C.	11.06
14° C.	11.91
15° C.	12.70
16° C.	13.64
17° C.	14.42
18° C.	15.36
19° C.	16.35
20° C.	17.39
21° C.	18.50
22° C.	19.66
23° C.	20.89
24° C.	22.18
25° C.	23.55
26° C.	25.00
27° C.	26.51
28° C.	28.10
29° C.	29.78
30° C.	31.55

20 CENTIMETRE SCALE IN MILLIMETRES.



Outlines of Industrial Chemistry

A TEXT-BOOK FOR STUDENTS

BY

FRANK HALL THORP, Ph.D.

Instructor in Industrial Chemistry in the Massachusetts Institute of Technology

Cloth. 8vo. Price \$3.50

A PARTIAL LIST OF CONTENTS

Part I. — INORGANIC INDUSTRIES. Introduction. Fuels. Water. Sulphur. Sulphuric Acid. Salt. Hydrochloric Acid and Sodium Sulphate. Soda Industry. Chlorine Industry. Nitric Acid. Ammonia. Potash Industry. Fertilizers. Lime, Cement, and Plaster of Paris. Glass. Ceramic Industries. Pigments. Bromine. Iodine. Phosphorus. Boric Acid. Arsenic Compounds. Water-Glass. Peroxides. Oxygen. Sulphates. Cyanides. Carbon Disulphide. Carbon Tetrachloride. Manganates and Permanganates.

Part II. — ORGANIC INDUSTRIES. Destructive Distillation of Wood. Destructive Distillation of Bones. Illuminating Gas. Coal Tar. Mineral Oils. Vegetable and Animal Oils, Fats, and Waxes. Soap. Candles. Glycerine. Essential Oils. Resins and Gums. Starch, Dextrin. Cane Sugar. Fermentation Industries. Explosives. Textile Industries. Paper. Leather. Glue.

Qualitative Chemical Analysis

BY

ARTHUR A. NOYES, Ph.D.

Assistant Professor of Chemistry in the Massachusetts Institute of Technology

8vo. Cloth. \$1.25 net

PROF. C. F. MABERY, Case School of Applied Science

"Having used the principal methods embodied in this work for more than twenty years, I can assert that they will give satisfactory results in teaching qualitative analysis with large as well as with small classes. The arrangement is excellent."

HERBERT R. MORLEY, Gilbert School, Winsted, Ct.

"This book we have used here for two years, and shall continue to do so indefinitely; since it was eminently satisfactory before, and is more so in its revised form."

DR. W. H. HIGBEE, Hamilton College

"I have used this work to some extent, and as a practical treatise on analytical chemistry find it to excel in just those points where other books of its kind usually fail, viz. in indicating precise methods of procedure as well as giving the rationale of the process employed."

PROF. W. P. BRADLEY, Wesleyan University

"It is the sort of manual I have been looking for, for use in my class, and I expect to give it a trial next year."

THE MACMILLAN COMPANY

66 Fifth Avenue, New York

An Introductory Course of Quantitative Chemical Analysis

WITH EXPLANATORY NOTES AND STOICHIOMETRICAL PROBLEMS

BY

HENRY P. TALBOT, Ph.D.

Professor of Analytical Chemistry in the Massachusetts Institute of Technology

THIRD EDITION, REVISED AND ENLARGED

8vo. Cloth. pp. 153. Price, \$1.50 net

PROF. C. F. MABERY, Case School of Applied Science

"It is an excellent work, carefully prepared, and on a plan that will supply a want in teaching quantitative analysis."

T. M. DROWN, President Lehigh University

"We find in Professor Talbot's Introductory Course of Chemical Quantitative Analysis something more than a book for local use. There is an idea running through it that is more or less new in books of this kind, and one which instructors will heartily welcome. There is an attempt to give the student not only the important and essential things which he should know in special cases, but what is of more importance, so to direct his thoughts toward his work that he shall grasp the fundamental ideas governing chemical analysis. In other words, the directions and explanations are such as the thoughtful and conscientious teacher would give to his pupil in the laboratory in endeavoring to make him think for himself intelligently."—*In the Journal of the American Chemical Society.*

The Practical Methods of Organic Chemistry

BY

LUDWIG GATTERMANN, Ph.D.

Professor in the University of Heidelberg

TRANSLATED BY

WILLIAM R. SHOBER, Ph.D.

Instructor in Organic Chemistry in Lehigh University

AUTHORISED TRANSLATION WITH NUMEROUS ILLUSTRATIONS.

12mo. Cloth. Price, \$1.60 net

"The selection and judgment throughout is excellent. The book is a most useful practical adjunct to any good text-book on Organic Chemistry."—*The Guardian.*

"This is a book that should be in the library of every teacher of Organic Chemistry, and one which will no doubt be of great value as a guide to students in their second year of organic chemistry. Its chief peculiarity and merit is in the great stress laid on practical laboratory work. . . . It is permanently a worker's guide."—*Pharmaceutical Review.*

"Dr. Gattermann is worthy of a translator, and Dr. Schober has done his work of translation well. Many really good books lose by being brought out in another tongue; 'Practical Methods of Organic Chemistry' loses none of its value and gains a new world of readers. . . . Text-books on Organic Chemistry are not too numerous, and a cheerful welcome is assured for Schober's Gattermann."

—*Science and Art of Mining, England.*

THE MACMILLAN COMPANY

66 Fifth Avenue, New York



THIS BOOK IS DUE ON THE LAST DATE
STAMPED BELOW

AN INITIAL FINE OF 25 CENTS
WILL BE ASSESSED FOR FAILURE TO RETURN
THIS BOOK ON THE DATE DUE. THE PENALTY
WILL INCREASE TO 50 CENTS ON THE FOURTH
DAY AND TO \$1.00 ON THE SEVENTH DAY
OVERDUE.

DEC 27 193

FEB 8 1940

FEB 22 1940

MAR 7 1940

22 Mar '59 BB

REC'D LD

MAR 25 1959

27 Aug 1959
REC'D LD

AUG 26 1959

LD 2.

QD45

HS

78217

